ODOR INTENSITY AND CHARACTERIZATION OF JET EXHAUST AND CHEMICAL ANALYTICAL MEASUREMENTS

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16. Abstract

Odor and chemical analyses were carried out on the exhaust samples from a J-57 combustor can operated over a range of inlet conditions, and with several fuel types and nozzle modifications. The odor characteristics and total intensity of odor for each exhaust were determined by a trained panel of NASA personnel over a range of dilutions to allow for a least-squares determination of the intensity at 1,000 to 1 dilutions. Analytical measures included the concentration of total hydrocarbons (by on-line flame ionization detector) and the concentrations of aromatic organic species and oxygenated organic species from collected samples which were taken concurrently. A correlation was found between the concentration of the odorous oxygenated fraction and the total intensity of aroma. Inlet operating conditions and nozzle modifications which increase the efficiency of combustion as measured by exhaust gas analyses reduce the odor intensity and the quantity of oxygenates in the exhaust. The type of fuel burned altered the intensity of odor in relation to the quantity of oxygenates produced and, in some instances, changed the odor character. A description of the equipment and procedures used is included.

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FOREWORD

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SYMBOLS

The following terms have been used frequently in the text and are defined for the convenience of the reader.

CRC   – Coordinating Research Council

EPA   – Environmental Protection Agency

NASA – National Aeronautics and Space Administration

T_i – Inlet air temperature, $^{\circ}C$

P_i – Inlet air pressure, atm.

V_r – Air reference velocity, m/sec

f/a   – Fuel/air ratio

TIA   – Total intensity of aroma. This is recorded on a 6-point scale in $\frac{1}{2}$ units with 3 being a maximum or strong odor level. Frequently tabulated as the value computed from the dose/response data at a 1000-to-1 dilution (1 ℓ/m^3) for exhaust or odorous fraction.

HRMS – High-resolution mass spectrometry.

R&DB – Rings and double bonds – a measure of the hydrogen unsaturation determined from the elemental composition by HRMS.

UV   – Ultraviolet absorption. The detector system used in the ALC recording optical density at 254 nm.

FID   – Flame ionization detector for measuring hydrocarbons.

ALC   – Analytical liquid chromatography – the basis for the instrumental method under development.

NA   – Preparative scale sampling on Chromosorb 102 500ℓ of exhaust sampled.

AN   – Analytical scale sampling on Chromosorb 102 – generally 30ℓ of exhaust sampled.

THC   – Total hydrocarbons as measured by FID.

TOE   – Total organic extract – the total organic exhaust extract of components recovered from the sample collection by solvent extraction.

LCP   – The paraffin fraction isolated from the preparative liquid chromatography procedure.

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LCA — The aromatic (oily kerosene) fraction isolated from the preparative liquid chromatography procedure. Concentration of aromatics measured by ALC.

LCO — The oxygenated (smoky-burnt) fraction isolated from the preparative liquid chromatography procedure. Concentration of oxygenates measured by ALC.

$\mu g/\ell$ — Concentration of exhaust species reported as $\mu g/\ell$ of exhaust; mg/m^3 or $mg/k\ell$ are equal to $\mu g/\ell$.

r^2 — A statistical measure of the % of information explained by the dependent variable.

σ — Standard error; a measure of the variability of the data.

I. SUMMARY

In March 1972, an experimental program was undertaken by Arthur D. Little, Inc., for Lewis Research Center, The National Aeronautics and Space Administration, to define the odor character and intensity of jet combustor exhaust, and to investigate the chemical relationships between odor intensity and odor character. It was planned to study and compare the sensory characteristics of exhaust from a combustor operated at a variety of inlet air conditions, with several different fuels and two or more nozzle modifications. The odor measurements were made by a panel of NASA personnel who had been trained by ADL to describe the odor characteristics and measure the odor strength. An odor test facility was designed and constructed to present exhaust samples over a range of concentrations. This technique for odor measurement summarizes the odor intensity of a given sample of exhaust by determining the least-squares line for the range of intensities and concentrations expressed as the logarithm. From this line, the total intensity of aroma for one liter of exhaust per cubic meter of dilution air can be calculated. This is the odor strength of each exhaust at a 1000-to-1 dilution.

Since preliminary experiments early in the program indicated that the techniques developed during our studies of diesel exhaust were applicable for sampling and analysis of jet combustor exhaust, little effort was expended in carrying out detailed identification studies of the very complex mixture of components which is the organic phase of jet combustor exhaust. However, analytical measurements based on the diesel exhaust work were taken at each experimental condition. These included the collection of the organic components from the exhaust by adsorption and their extraction from the adsorbent with solvent. The total organic extract was separated and quantified using analytical liquid chromatography to determine the aromatic components, representing the fuel, and the oxygenated species, representing the more odorous partial combustion products. In addition, a more detailed analysis of the relative distribution of various chemical species, based on their chemical compositions, was conducted by means of high-resolution mass spectrometry. This analysis was undertaken to assure that the chemistry of the odorants was consistent with that previously determined for diesel exhaust. In addition, it was felt that such information might provide a means for differentiating between exhausts of significantly different odor types.

Finally, Standard exhaust emission measurements were taken during the test run with on-line monitoring equipment operated by NASA personnel. These measurements monitored the inlet and exit operating parameters, including the concentrations of total hydrocarbons, CO, CO_2, NO, and NO_x as well as the exhaust temperature profile. All of this information was automatically recorded in the NASA data acquisition system. From these data the thermal efficiency of combustion (eta) and the efficiency based on exhaust composition (CEFF) were calculated.

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As a result of these studies, a significant correlation was found between the total intensity of aroma for combustor exhaust and the logarithm of the partially oxygenated components, which extends over all operating conditions, nozzle modifications, and fuel types. Similar semi-logarithmic correlations were found with the concentration of the aromatic components and, to a lesser degree, with the total hydrocarbons as measured by the on-line system. In addition, an inverse correlation was found between the logarithm of the efficiency of combustor operation, as measured by exhaust gas analyses, and the intensity of the exhaust odor. At high-efficiency operation, the odor level is significantly lower than at low-efficiency operation. Minimum odor levels were observed when the efficiency fell in the 99.8 to 99.9% range. Combustor modifications which improved the efficiency of combustion (such as the air assist nozzle at low fuel flows and inlet operating conditions, or the Simplex nozzle at high fuel flows and inlet air conditions) significantly reduced the odor intensity as well as the concentration of oxygenates in the exhaust.

Different fuels were also found to affect the intensity of odor. This variation in intensity is proportional to the oxygenates produced. Therefore, although the intensity of odor at all operating conditions proved to be substantially lower with natural gas as the fuel, the concentrations of oxygenates measured were proportionately low as well. A reduction of odor was also observed when isooctane was used as a fuel, again in relationship to the quantity of oxygenated species produced.

Differences were noted in the character of the exhaust, particularly between fuels. Most noticeable were the characteristics associated with isooctane, which was described as "burnt sweet" rather than "smoky burnt", and was less pungent. When natural gas was burned the levels were substantially lower in odor intensity, but also were associated with burnt sugar descriptors and, at one condition, with irritants such as acrolein.

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II. INTRODUCTION

During the past twelve months, Arthur D. Little, Inc., has carried out
a study of the odor characteristics and their intensities from jet com-
bustor exhaust gases under contract to, and in conjunction with, NASA-
Lewis Research Center. NASA-Lewis provided the test facility, operators,
and on-line monitoring equipment of the exhaust gases. In addition, per-
sonnel from NASA-Lewis were trained to carry out the odor measurements
under the direction of the ADL staff. Analytical aspects of the program
were carried out by the ADL staff, both at NASA-Lewis and in Cambridge,
Massachusetts, using methodology developed primarily as a result of ADL's
work for the Coordinating Research Council (CRC) and the Environmental
Protection Agency (EPA) on diesel exhaust odor.[1,2,3,4] Although con-
siderable effort has been expended on analyzing the odors of diesel ex-
haust during the past 10 years, including work at Southwest Research
Institute, Illinois Institute of Technological Research Institute, Scott
Laboratories, and the Bureau of Mines, to mention a few, no detailed
studies of jet combustor or jet turbine odor levels have been conducted.

The program was originally outlined in three tasks: (1) the orientation
and design phase, (2) the construction of the odor test facility and
training of an odor panel, and (3) the examination of the effects of com-
bustor variables on the intensity and odor character of the exhausts. This
program resulted in the development of useful and significant correlations
between levels of certain organic species and the intensities of odor, as
well as between odor intensity and the efficiency of combustor operation.
The success of the program was due, in large part, to the technology and
expertise developed in the course of the five years of work with CRC/EPA.

Objective

The statement of work of the original program had dictated a study program
to define the chemical nature of the odorous components of jet combustor ex-
haust under various operating conditions, and to measure the intensity of odor
and its character. Since a significant amount of the analytical information
needed had been developed in the first three years of our study of diesel
exhaust, repetition of the detailed analytical work did not appear to be
warranted. The technology had been developed to a point at which certain
chemical measures which correlated with diesel exhaust odor intensity had
been taken and defined. The study was directed at determining that these
measures also related to jet combustor odor levels. The objective of the
sensory analysis program was to obtain measures of intensity based on a
variable dose presentation of the exhaust, to relate to concurrent measures
of the concentrations of odorous components. Additionally, the panel was
asked to describe the character of the odor, particularly as it differed
between test variables. Samples of sufficient size were taken so that

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odorant presentations could be made at subsequent times and more detailed analyses could be made of the kinds and relative concentrations of chemical species present.

The objective of the experimental program, therefore, was to (1) measure the odor intensity of jet combustor exhaust under a wide variety of operating conditions, (2) characterize the odor types associated with either fuel or operating conditions, (3) measure chemical concentrations in the exhaust to correlate with the intensity measure, and (4) probe the variation in the chemical composition of the complex mixture represented in the oxygenate fraction derived from jet combustor exhaust.

Experimental Approach

The experimental studies, aside from the tasks outlined in the Work Statement, were carried out in three phases. An initial exploratory run was carried out in the first month of the program to demonstrate the applicability of the techniques developed in ADL's earlier studies of diesel exhaust to the collection and analysis of jet combustor exhaust. When these techniques proved to be satisfactory, a survey of the extremes of the engine operating parameters using ASTM-A1 fuel was carried out during the months of July and August. From this work, it appeared that essentially two levels of odor could be produced by the combustor: a higher level of odor associated with low-efficiency operation, and a lower level of odor associated with high-efficiency operation. Therefore, during the third phase of the experimental work (which represented the bulk of the effort) three combustor-inlet conditions were examined with a variety of fuels and combustor modifications. At the conclusion of the program, three additional inlet air conditions of potentially lower operating efficiency were examined. During the third phase of the program, each variable was replicated on separate days in its entirety, and for each test point, duplicate samples were taken for analysis while odor measurements were made both in ordered and random presentation.

The inlet air conditions in the survey program were varied from $10-240^{\circ}C$ inlet air temperature, 1-4 atm. inlet air pressure, 15-36 m/sec air reference velocity, and 0.008 to 0.016 fuel/air ratio.

Exhaust samples were presented to a trained panel of NASA personnel and concurrently sampled for chemical analysis. A schematic diagram of the process appears in Figure 1.

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Combustor Exhaust — Hot Filter — Chromosorb Trap — Wet Test Meter

Odor Panel Analyses

On-line Analyses

Hydrocarbons
CO
CO_2
NO
NO_x

Preparative NA (500ℓ)

Analytical AN (30ℓ)

Analytical Liquid Chromatography ALC/UV (all samples)

Solvent Extract TOE

Preparative Scale Liquid Chromatography

Paraffins LCP

No Odor

Aromatics LCA

Odor Mass Spectrometry

Oxygenates LCO

Odor High Resolution Mass Spectrometry

FIGURE 1   SCHEMATIC OF THE ANALYTICAL PROCESS

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III. EXPERIMENTAL PROGRAM

The purpose of the experimental program was to define the odor character and intensity of jet combustor exhaust and to understand the chemistry that contributes to its odor character and intensity. Such a program requires detailed and reliable odor data which can be correlated with analytical measurements of sufficient sensitivity to determine the concentrations of those components that influence the odor sense. To a large extent, the analytical side of the program had been developed as a result of the several years of work performed by ADL on diesel exhaust odor under the sponsorship of CRC/EPA, and it remained to automate and improve the analytical system to provide reliable information of the concentrations of components in the μg/ℓ range. In the diesel exhaust program, ADL's staff of highly trained and experienced odor analysts was used to develop the analytical correlations and an understanding of the chemistry of diesel exhaust odor. In the present program, NASA personnel were used to implement the odor measurements. This required training and indoctrinating a group of volunteers to provide the necessary information.

The study of odors is, at best, a difficult problem in that different chemicals and combinations of chemicals may be perceived as different odor characteristics or odor types. However, an individual's memory for odor character is good, and he can be trained to recognize and differentiate between thousands of different odor types. Odorant chemicals at different concentrations produce different strengths of perceived odor. This is referred to as the odor intensity. It is well recognized that the perceived sensation is a logarithmic function of the concentration,[5] and it is important to remember this logarithmic relationship to odor strength in understanding the requirements of an analytical program. In addition, odorants evoke what is frequently termed an hedonic response; i.e., people either like or dislike the odor type. This influences the way an individual responds to the intensity of odor, and thus the relative strength of an odor that is reported may not reflect a difference in concentration. For example, a low intensity of butyl mercaptan may be judged and reported to be strong because of the unpleasant character of the skunk odor. A similarly low level of citral may be judged to be pleasant, and therefore, a slight intensity would then be recorded, whereas the actual odor strengths of the two compounds could be deemed approximately the same. One function of the odor training program is to minimize the effects of such like/dislike judgments in reporting intensities.

In measuring odor it is essential to have a presentation system which eliminates distractions, either from noise or interruptions, as well as extraneous odors. The system must also provide for reproducibly controlling the concentration of the odorant dose presented to the panel.

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Much odor intensity information has been obtained on the basis of presentation of the odorant at a single concentration, using an intensity scale as the means of scoring differences. This technique has two problems: first, it can be shown that at both low and high concentrations the discrimination between changes in concentration is less precise than the intensity discrimination in the middle portion of the intensity range; and, second, by obtaining a series of responses from a set of concentrations, the intensity measure can take into account the influence of the change in perceived intensity with increasing concentrations. The series of measurements can then be summarized into a single data point by means of a least-squares regression analysis of the dose response curve for the individual panel members or the panel together. This results in an equation of the form TIA = a + b logC, where C is exhaust concentration in ℓ/m^3. The intercept (a) is the TIA, where C=1 ℓ/m^3 and (b) is the slope of the dose/response relationship.

A small panel was trained to make odor measurements at five test concentrations, usually from 0.15-3.6 ℓ/m^3, with each test about double the previous concentration.

A regression analysis has least variability in the middle portion of the data. It was, therefore, mathematically correct and convenient to select a value to represent the total intensity of exhaust as the TIA calculated for exhaust at a concentration of 1 ℓ/m^3. Although the concentration is not precisely in the center of the dose/response data, it is close to it. Since all of the analytical measurements are recorded as $\mu g/\ell$ of exhaust, and since the TIA is calculated for 1 liter of exhaust per cubic meter of dilution air, the analytical concentration can be directly substituted to give the concentration of odor-active components as $\mu g/m^3$ of dilution air. (This is a dilution of about 1 ppb on a volume basis.)

The following sections of the report describe the efforts related to the construction of an odor test facility at NASA-Lewis; panel selection and methodology of odor measurements, the exhaust sampling and chemical analytical procedures for measurement of odorous species, and finally the combustor operation and test program.

Odor Test Facility – Design and Construction

The odor test facility consists of a chamber approximately 2.4 meters wide, 3.6 meters long, and 2.4 meters high, with a plenum chamber located on the rear wall in which exhaust can be diluted to the desired concentration with activated carbon-treated air, and exhausted to the outside without contaminating the air in the vicinity of the test chamber. Figure 2 presents the layout and flow patterns of the facility, while Figure 3 shows the orientation of equipment. To provide low ambient
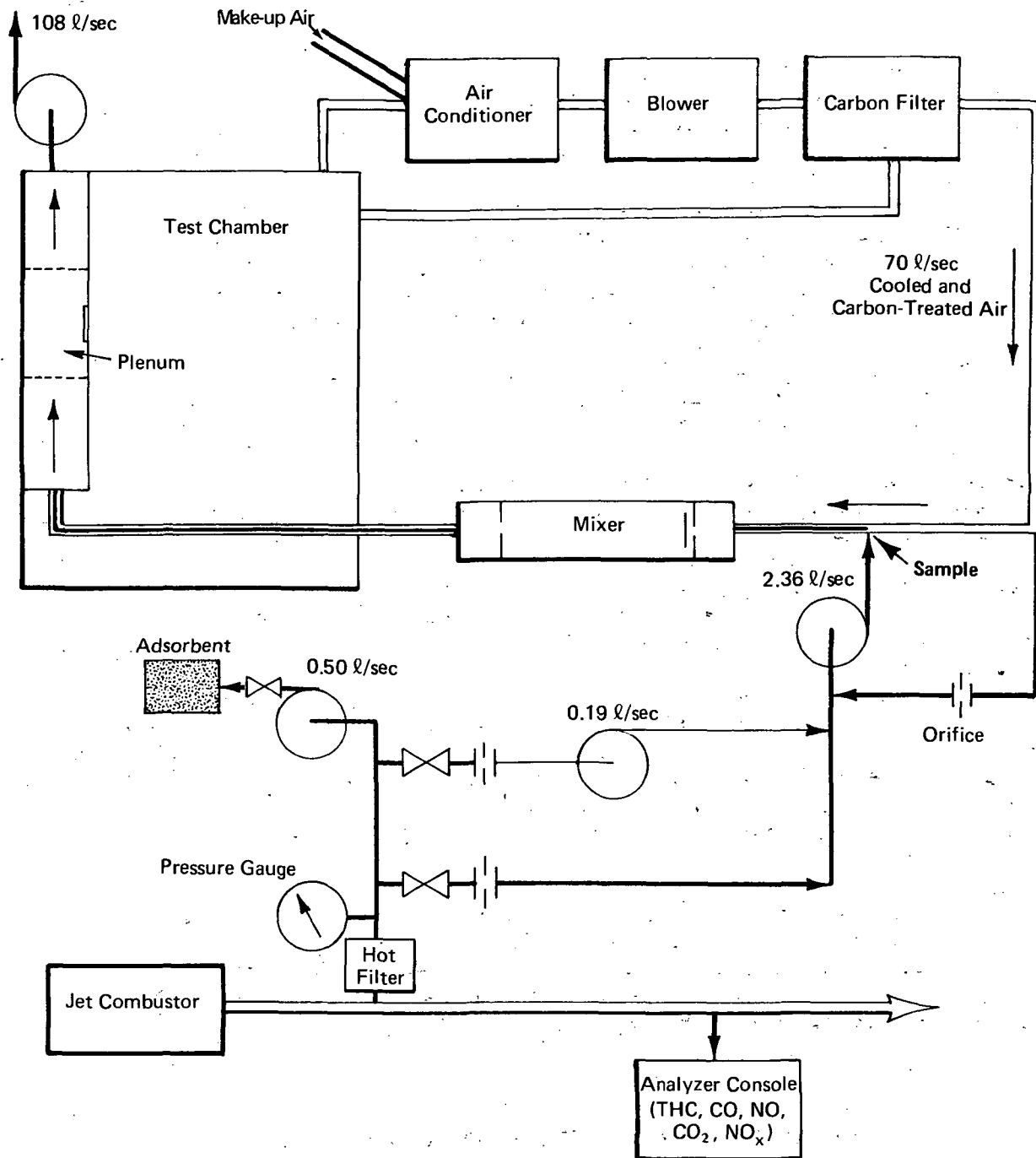
FIGURE 2   SCHEMATIC ARRANGEMENT OF EQUIPMENT
FOR NASA ODOR TEST FACILITY

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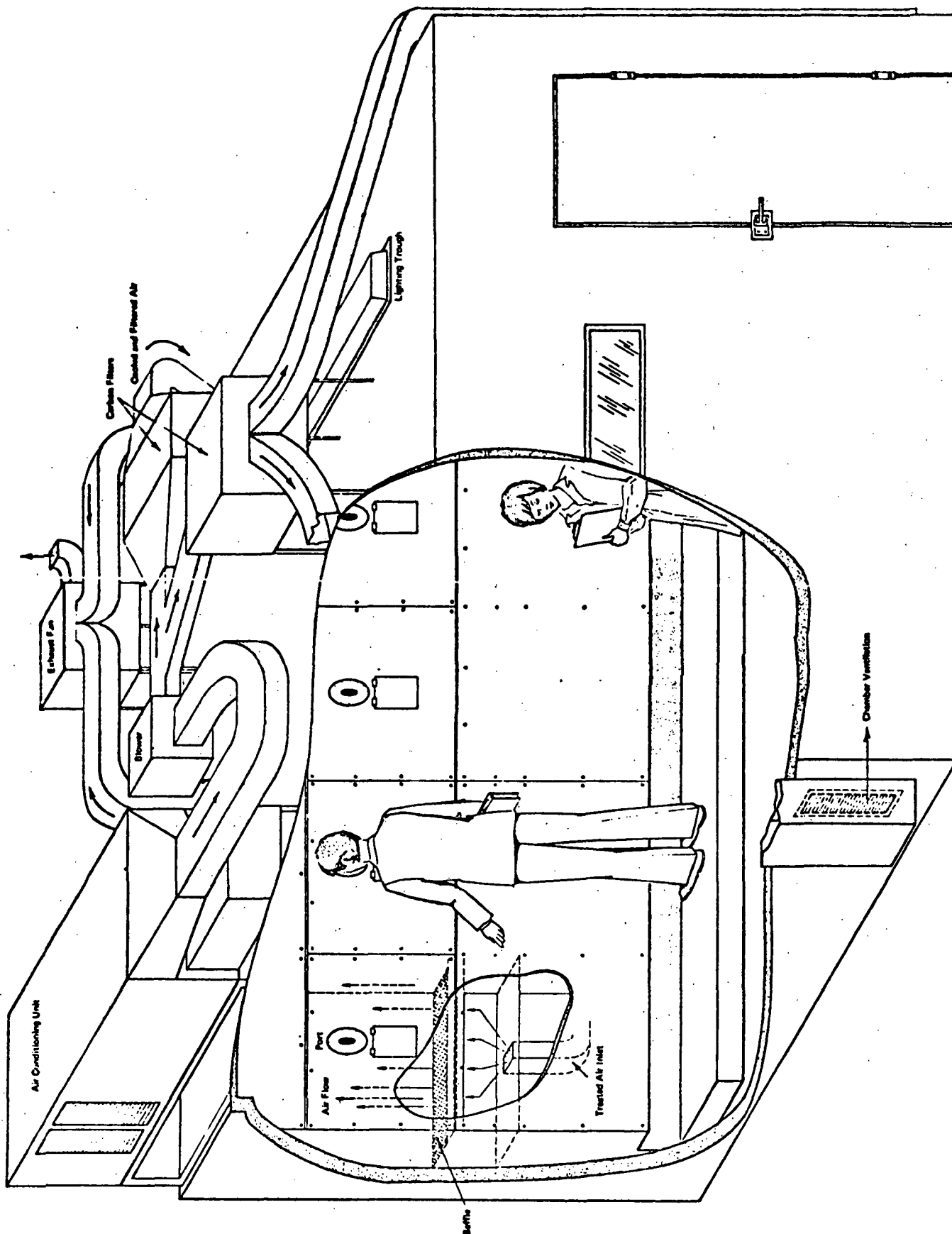
FIGURE 3

CUTAWAY VIEW OF NASA ODOR TEST FACILITY

Air Conditioning Unit

Exhaust Fan

Carbon Filters

Cooled and Filtered Air

Lighting Trough

Blower

Air Flow

Port

Treated Air Inlet

Baffle

Chamber Ventilation

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odor in the test facility and for the comfort of panel members during the analysis, the air is cooled and carbon-treated for both the test chamber and for the plenum. The air from the test chamber is recycled with make-up air added at the intake to the air conditioner. The test chamber is maintained at a positive pressure with respect to the plenum chamber to produce flow into the exhaust stream when the sniffing ports are open. The sniffing ports (Figure 4) located side by side along the rear wall of the test chamber, are normally sealed with an aluminum plate and a deodorized rubber gasket material. The hole which has been cut through the gasket is shaped to accommodate the analyst's face so that the nose is in the air stream while air flow around the face is minimized. The flow of air in the plenum chamber is from bottom to top at linear flow rate found to be approximately 15 ℓ/sec. This air flow is not uncomfortable for the analyst, and variability in flow rate does not affect the concentration of exhaust in the air stream.

During the test day, air is continuously cycled through the test chamber and through the plenum chamber, from which it is exhausted at about 108 ℓ/sec. The recirculated air acts to maintain a low odor level in the test chamber. The exhaust system removes the odorized sample and extraneous odors from the plenum chamber and part of the air from the testroom to minimize the build-up of odor at the sniffing stations. Little build-up of odor was observed in the test chamber, and with air conditioning the facility remained quite operable, even with 30°to 35°C ambient air temperatures. Total air flow to the plenum chamber was measured at 70 ℓ/sec, using a vane-type anemometer, which was approximately the diameter of the input duct.

A small constant flow of this air is taken for dilution air by means of a 2.36 ℓ/sec metal bellows pump. To produce a range of exhaust concentrations at dilutions appropriate for odor analysis, a smaller, 0.19 ℓ/sec, sampling pump takes exhaust through one of five orifices to give five predetermined and controlled flows of exhaust. Figure 5 shows the exhaust sampling and dilution system. The diluted exhaust was mixed with the total air flow to the plenum by means of an orifice-type mixing tube. This can be seen in the rear view photograph of the test chamber in Figure 6. The delivery rate of the 2.36 ℓ/sec pump was found to be 1.98 ℓ/sec at atmospheric inlet pressure, which gives a constant 35-to-1 dilution for all samples.

Concentration ratios, obtained for the five orifices presently in use, are given in Table 1.

FIGURE 4   NASA PANEL INSIDE ODOR TEST FACILITY

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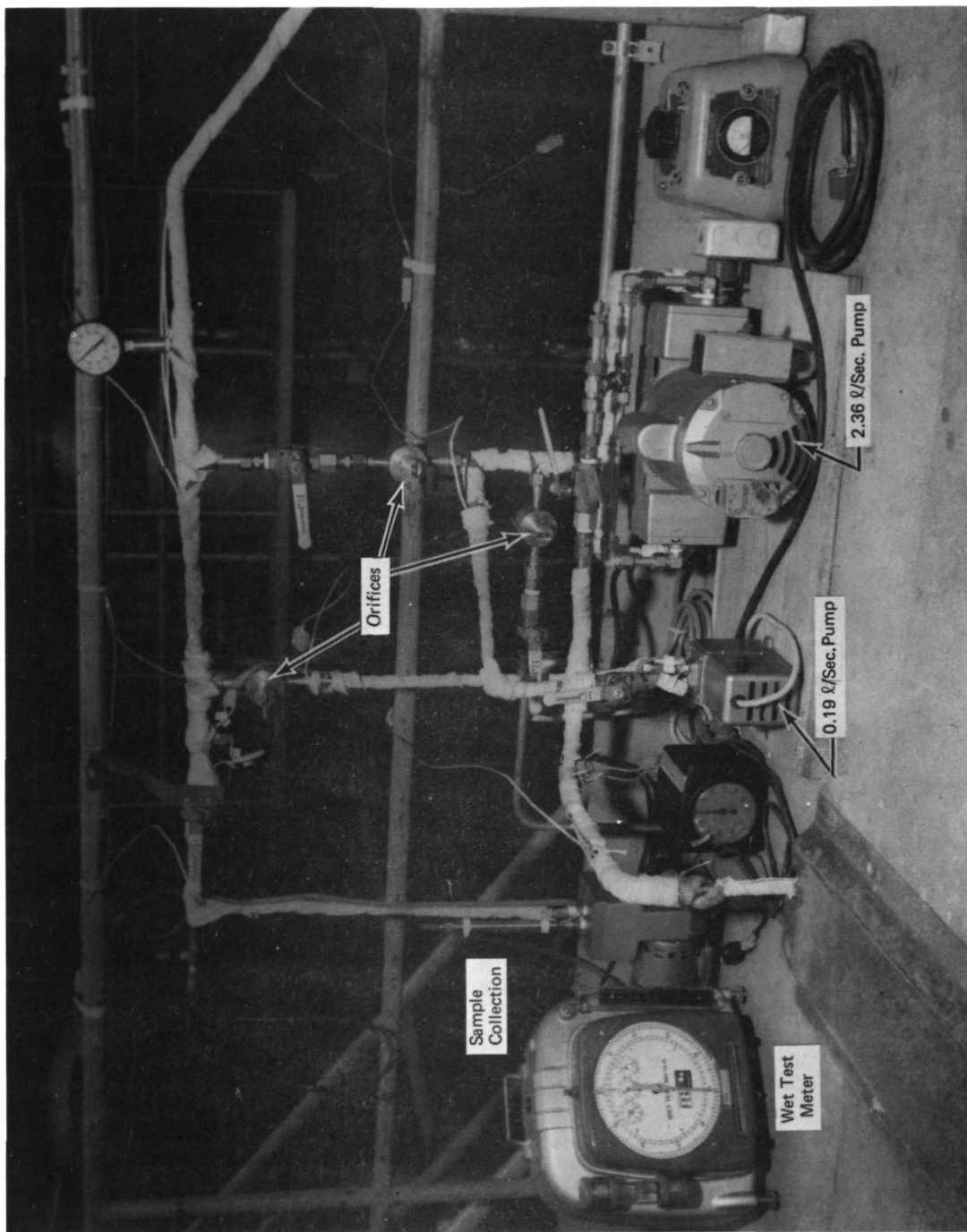
FIGURE 5   EXHAUST SAMPLING AND DILUTION SYSTEM

Labels in figure: 2.36 ℓ/Sec. Pump; 0.19 ℓ/Sec. Pump; Orifices; Sample Collection; Wet Test Meter

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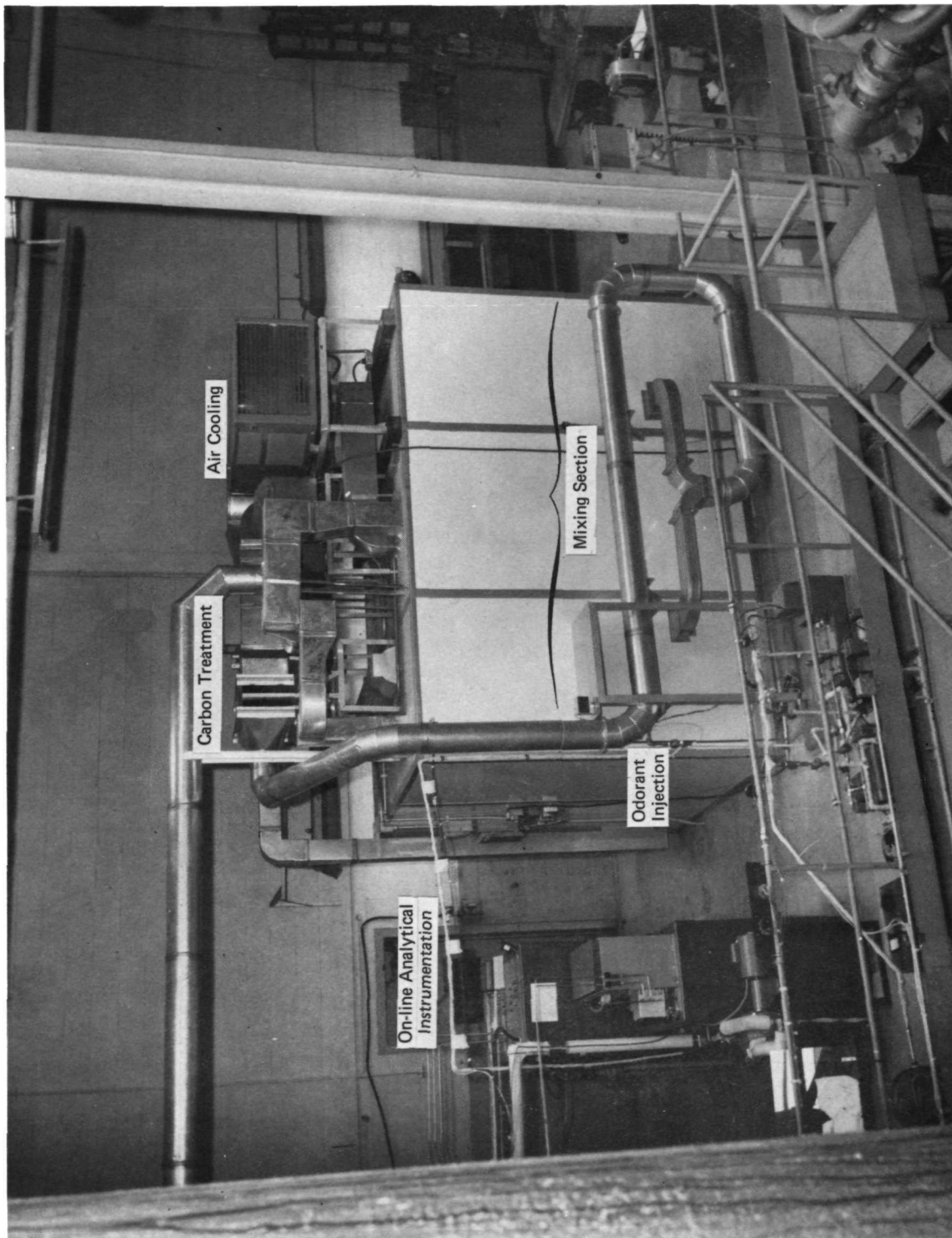
FIGURE 6   REAR VIEW OF ODOR TEST FACILITY

Air Cooling

Carbon Treatment

Mixing Section

Odorant Injection

On-line Analytical Instrumentation

TABLE 1

EXHAUST CONCENTRATIONS FOR ODOR TESTS

Test Condition	Total Dilution	Exhaust Concentration ℓ/m^3
T_1	6600/1	0.15
T_2	3300/1	0.30
T_3	1600/1	0.62
T_4	700/1	1.4
T_5	280/1	3.6

The system was designed to produce five concentrations, each one approximately double the previous concentration. As can be observed from the dilutions calculated from CO measurements at the smaller diameters, the effect is that of a critical orifice, and the ratio holds fairly well. At the two higher concentrations, the doubling is not exact but this is taken into consideration in the mathematical analysis of the data, although it introduces some bias.

To obtain higher concentrations of exhaust, the system is equipped with two sets of orifices to control the relative flows of exhaust and dilution air to the 2.36 ℓ/sec pump without the use of the 0.19 ℓ/sec pump. Dilution ratios in the range of 200/1 to 35/1 can be produced by selecting the appropriate pair of orifices for the exhaust sampling line and the diluting air stream (Table 2).

TABLE 2

EXHAUST CONCENTRATIONS FOR HIGH DOSE ODOR TESTS

Test Condition	Dilution	Exhaust Concentrations ℓ/m^3
T_{1H}	180/1	5.6
T_{2H}	110/1	9.0
T_{3H}	80/1	12.5
T_{4H}	71/1	14.0
T_{5H}	36/1	28.0

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To assure complete odor removal, a standard three-minute interval is waited between all tests. Experience has indicated that it requires approximately 20 seconds for the mixed exhaust and dilution air sample to reach the sniffing ports after the valve has been opened from the exhaust line to the 0.19 ℓ/sec pump. The odor test itself requires something less than 30 seconds for all four panel members to make their analyses and record odor intensity and character.

A second sampling system has been developed to permit the injection of diluted samples of the total organic extract (TOE) collected in the preparative scale sampling procedure. A Harvard syringe pump (Figure 7) is located so that the syringe needles can be inserted into a heated segment of a 1.27 cm stainless-steel line on the outlet of the 2.36 ℓ/sec pump (or later a 0.50 ℓ/sec pump). A uniform flow of a solution of the TOE at varying rates can be perfused into the air stream during a timed sequence. At No. 6 gear setting, the pump is found to deliver a maximum of 2 μℓ/sec. At this gear setting the feed rate can be continuously varied down to less than 10% (0.2 μℓ/sec). Thus, the concentration range of raw exhaust presented can be approximated by controlling the dilution of the TOE in a low odorous solvent, such as nonane.

Panel Selection and Odor Measurement

A major portion of the program depended on the development of reliable odor information, both descriptive as to type and quantitative as to strength. Since odor is the sensory response to certain chemicals, called odorants, it is necessary to use human subjects and train them to be objective in their measurements. Therefore, one day was spent in selecting eight interested and capable individuals from a group of sixteen volunteers from Lewis Research Center.

Panel Orientation

The eight panelists were given three days of training which included practice in the identification of different odor types related to diesel exhaust and scaling the odor strength perceived with different concentrations of odorants. Reference standards for use in describing odor types were taken from ADL's library of odorants as well as mixtures of chemicals and exhaust extracts derived from the diesel exhaust studies.

The intensity scale used by the panel is a simple six-point scale described in Methodology of the Flavor Profile.[6] It represents an individual's ability to discriminate between slight, moderate and strong odor levels, which are designated as 1, 2, and 3. With training, the panel members can estimate intermediate levels with reasonable reliability, and these

Harvard Syringe Pump
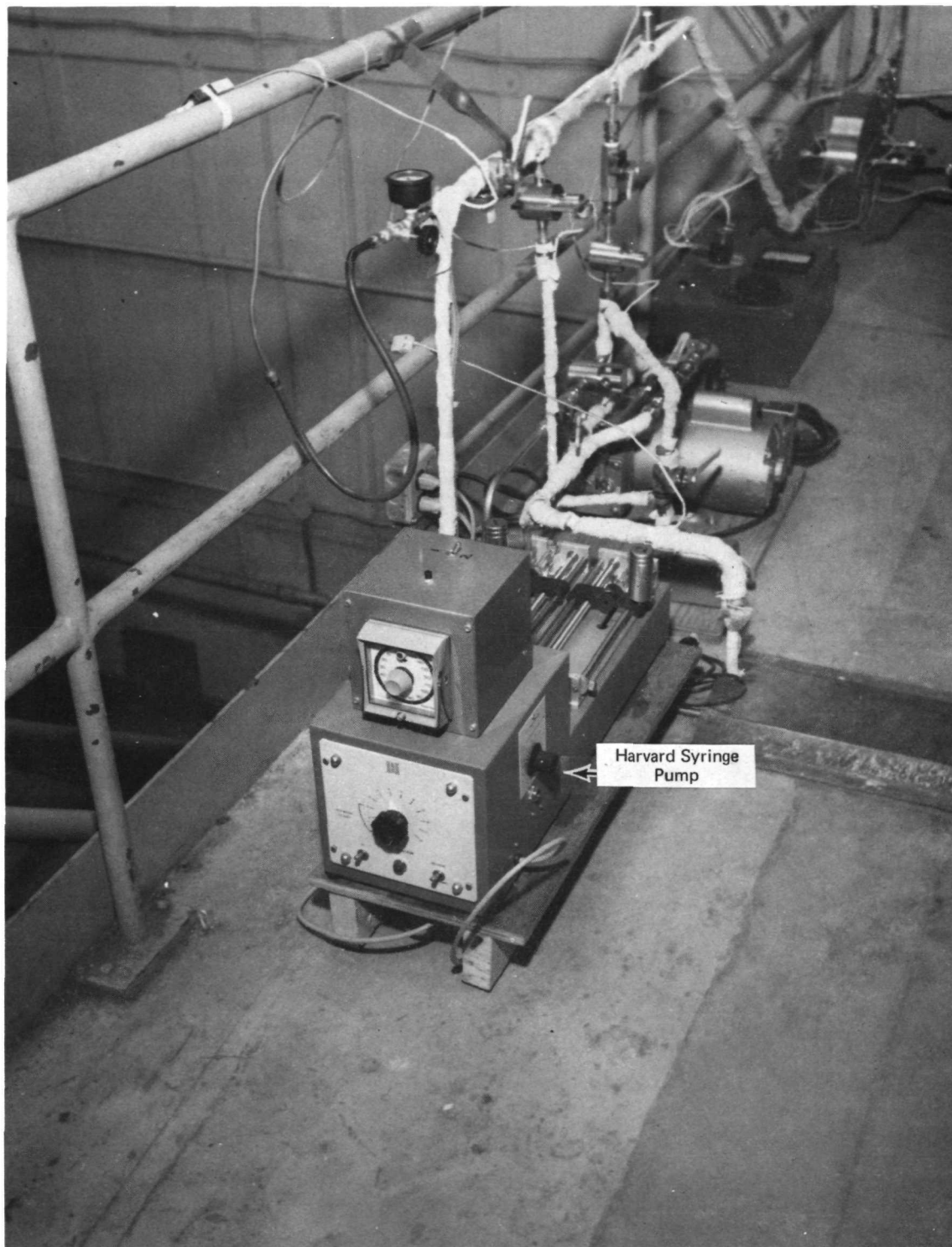
FIGURE 7   ODORIZATION SYSTEM FOR ODORANT SOLUTIONS

are designated as ½ integers. Our experience indicates that the concentration difference between slight and moderate intensities may be as much as a tenfold increase in concentration. It is possible for an individual to reproducibly measure substantially smaller differences in intensity, but only where direct comparisons can be made without sensory fatigue.

Although the ability to rate intensity developed rather rapidly, the development of descriptive vocabulary was a continuing process throughout the program. The performance of the panel, even during the odor survey which was their first task, was good and the results were uniformly of value in understanding the effect of operating variables on odor.

Odorant Presentation

Based on a number of odor studies carried out at ADL, and the primary concern for determining reliable intensity data, exhaust from each test was presented in a series of five concentrations, each approximately twice the preceding level. Each panelist independently recorded the total intensity of aroma (TIA) and the primary odor character notes. This required a minimum number of sniffs (2-3) and less than 30 seconds at each concentration. After completion of one dose/response series in order of increasing concentration, the same doses were presented in random order. At least three and preferably four NASA panelists participated at each session with one ADL staff member acting as panel leader.

Data Analysis

The TIA data is most easily summarized by calculating the least-squares line for TIA vs log of the exhaust concentration in liters per cubic meter. The TIA intercept, i.e., the TIA calculated for 1 liter of exhaust per cubic meter of dilution air, represents the information from 20 to 25 observations for both the ordered and the random sets. These sets were kept separate so that the effects of the two different modes of presentation could be assessed.

The summarization of the descriptive information requires some interpretation and weighting of individual's experience and vocabulary. Since descriptive information is recorded for all presentations, but usually in greater detail for the ordered series, the descriptive odor profiles present the most frequently used reference terms and eliminate wording that is redundant. Of primary interest is the description of combustion components "smoky", "burnt", "sooty", and "oxidized oil", as well as fuel related odor characteristics "solventy", "kerosene", "naphthalene", "oily". In addition, the irritation factors are tabulated. The profile summary represents the odor characteristics at an intermediary concentration rather than the high or low doses.

Exhaust Sampling and Chemical Analyses*

Sampling Methods

Jet combustor exhaust samples from which the particulates have been removed by filtration through a heated fiberglass filter are collected on preparative traps and analytical traps. The preparative trap (Figure 8) contains 10 g of prewashed Chromosorb® 102 (60/80 mesh, Johns Manville) and is used for the collection of 500ℓ of exhaust. The analytical traps are used for the collection of small amounts of exhaust, e.g., 30ℓ . These are made from a 7.5 cm piece of 0.8 cm O.D. stainless-steel tubing fitted at each end with 0.4 x 0.8 cm Swagelok® reducing union and hold about 1 g of Chromosorb® 102.

The organic components of exhaust adsorbed by the Chromosorb® are subsequently extracted by allowing freshly distilled chromatographic-grade pentane to slowly percolate into the Chromosorb® bed at the rate of approximately 0.5 mℓ/min. The solvent flow is countercurrent to the direction of exhaust flow to the trap during sample collection. The extracts so derived are known as the total organic extract (TOE). The TOE fractions contain essentially all of the odor. Ten mℓ of pentane effluent are required for the complete extraction of the exhaust samples from the preparative traps, while 1 mℓ of effluent is sufficient for the analytical traps. Therefore, the extract from the preparative trap is equivalent to 0.05ℓ of exhaust/μℓ of solution, while the extract from the analytical trap is equivalent to 0.03ℓ of exhaust/μℓ of solution.

The TOE samples obtained from the preparative traps, which represent a fairly large volume of exhaust, are suitable for odor studies as well as the total analytical methodology consisting of gas chromatographic (GC) analysis, analytical liquid chromatography/ultra-violet analysis (ALC/UV), preparative column chromatography for the isolation of the aromatics (LCA) and oxygenates (LCO) fractions, and high-resolution mass spectrometry (HRMS) analysis. The small volume of TOE sample from the analytical or preparative sampler is sufficient only for ALC/UV analysis and GC analysis.

* See Figure 1 for process flow.
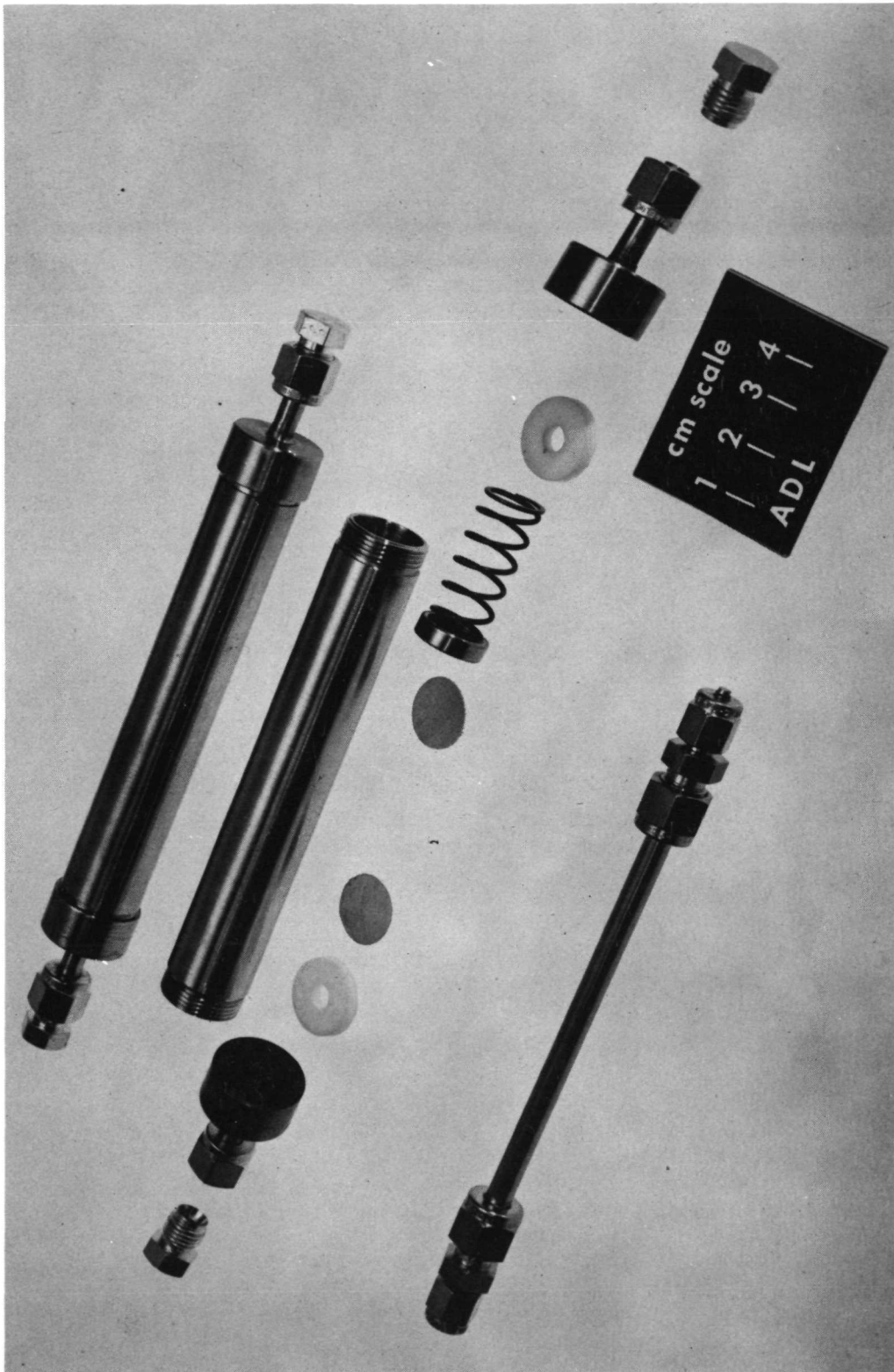
FIGURE 8   EXHAUST SAMPLING TRAPS

Analytical Methods

An approximation of the total hydrocarbon content of the TOE samples can be made by low-resolution gas chromatography analysis, i.e., a ballistic program from ambient temperature to 205°C on a short 10% OV-1 column, 46.0 cm x 0.35 cm stainless steel. (A Perkin-Elmer Model 900 GC unit with a flame ionization detector was used for our work.) A 1% solution of fuel oil has been used as the calibration standard. The same mode can be used to analyze the aromatic and oxygenate fractions to determine their relative mass content. This analysis gives the μg abundance of each fraction per ℓ of exhaust.

Analytical liquid chromatography - The ALC method is based on the use of newly available commercial components assembled as shown schematically in Figure 9. The following components are used:

> (1) Two solvent reservoirs, hydrocarbon and alcohol
>
> (2) Positive displacement pump
>
> (3) A silicon septum sealed injector
>
> (4) A 30-cm x 0.6-cm O.D. glass column packed with a silica gel-type support-Corasil II (Waters Associates, 37-50 microns)
>
> (5) Alcohol solvent injection valve (50 μℓ)
>
> (6) Ultraviolet absorbance detector and amplifier, detecting wavelength 254 nm
>
> (7) Recorder

The unit operates essentially the same as a gas chromatograph, except in the liquid phase. A solvent flow is established through the column (1 mℓ/min), a sample is injected with a syringe, and the separated components which absorb ultraviolet light at 254 nm are recorded as they elute from the column.

The aromatics (LCA) are eluted with the solvent front in the 35% CH_2CL_2/hexane and the oxygenates (LCO) are eluted after injection of 50 μℓ of methyl alcohol. The resulting chromatogram is shown in Figure 10.

The aromatics (LCA of Figure 10) represent the oily kerosene odor notes, while the oxygenates (LCO) represent the smoky-burnt character.

It was established that optical density varies linearly with sample concentration for both aromatics and oxygenate peaks.[7] Thus, it becomes a simple matter to determine the aromatic and oxygenate content of every exhaust sample by injecting 10 μℓ aliquots (ca equivalent to 0.3 to 0.5 ℓ of exhaust) of TOE samples.

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Signal Conditioning and Control Logic

2 Pen Recorder

A — Check Valve
B — Check Valve

.6 cm x 30 cm ss Column

To Waste

B

254 nm UV Detector

Septum Sample Injector

Solenoid

To Waste

Methanol

50 ul Loop Injection Valve

Gauge

Pulse Damper

A

Solvent Reservoirs

Positive Displacement Mini Pump

35% Methylene Chloride in Hexane

– 21 –
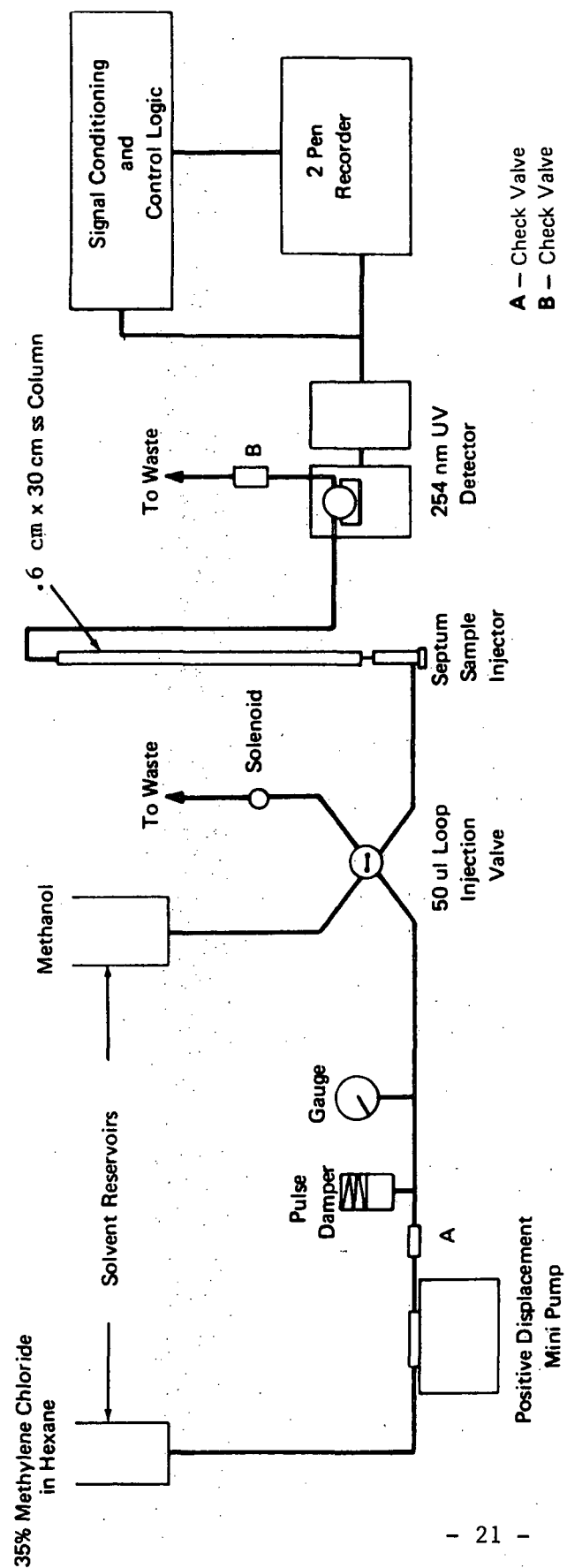
FIGURE 9   ALC RESEARCH PROTOTYPE SYSTEM
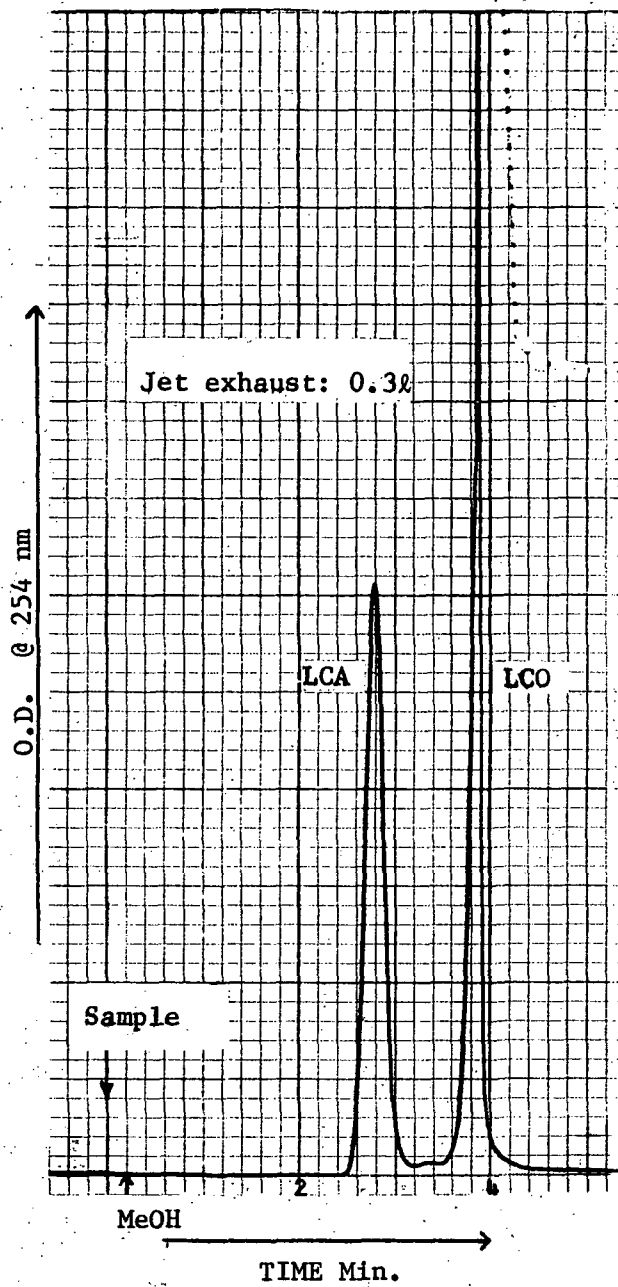
FIGURE 10 ALC/UV CHROMATOGRAM OF JET EXHAUST TOE SAMPLE

Preparative scale liquid column chromatography - Large volumes (500 ℓ)
of exhaust can be resolved into the three characteristic fractions --
paraffins, aromatics, and oxygenates -- by adsorption chromatography on
silica gel. These three separated fractions can be submitted for HRMS
analysis or odor studies.

A glass column, 30 cm x 1 cm I.D., is dry-packed with 6.5 gm of freshly
activated silica gel (60-200 mesh, Grade 950). Pentane (distilled chroma-
tographic grade) is passed through the packed column until the silica bed
is homogeneous and free of trapped air bubbles. Then the TOE sample
(in 10 mℓ of pentane) is added to the column and eluted as shown in Table 3
to give the desired fractions.

TABLE 3

PREPARATIVE COLUMN CHROMATOGRAPHY OF EXHAUST SAMPLES

Fraction	Solvent + Effluent Volume	Components Eluted	Odor Characteristic
LCP	Pentane, 25 mℓ	Paraffins	Odorless
LCA	Methylene chloride, 10 mℓ	Aromatics	Oily kerosene
LCO	Methanol, 25% $\}$ -10 mℓ Methylene chloride, 75%	Oxygenates	Smoky burnt

Mass spectrometry - To better define the chemistry of the LCA and LCO
fractions derived from preparative-scale separation, it was possible
to make use of the analytical capabilities of mass spectrometry in either
the low - (for LCA) or high - (for LCO) resolution modes. During the
studies of diesel exhaust,[3] we were able to show that matrix analysis
of the low-resolution mass spectrum of the LCA fraction containing the
oily-kerosene odor would provide a measure of the amount of alkyl benzenes
and indans/tetralins in that sample, as well as the more abundant naphtha-
lenes. Further, an indication was obtained that the amount of indans/
tetralins injected into the odor test room, as computed from the analytical
data, did relate to the kerosene odor intensity. To a lesser extent, the
alkyl-benzene concentration also correlated with the oily odor intensity
of those fractions.

We also suggested that, although the oxygenates' smoky-burnt odor fraction
was much more complicated, a comparable type of analysis might be possible
utilizing data obtained from the complete high-resolution mass spectrum
of the LCO liquid chromatography fraction. It is important to remember
that we do not know with the certainty that was established for the
oily-kerosene odor fraction just which groups of oxygenated species we
prefer to measure to reflect the odor of that sample. Therefore, an

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analytical method for the oxygenate fraction should summarize all of the chemical species concentration data where possible, but leave a maximum possibility for examining the data in several combinations.

The oxygenates' analysis then is a means of representing, on an internally consistent basis, the different types of chemical groups present in the LCO fraction which were identified as odorous species. The matrix (Table 4) does not represent the actual percent concentration of these groups in the sample, but rather a relative abundance influenced by different instrument response to different species. The system does provide an efficient means of comparing samples and searching for odor-significant differences. The R+DB value is a definition of chemical class based on hydrogen unsaturation, while columns O_1 and O_2 indicate the relative amounts of each class found with that number of oxygens. Thus, R+DB $1/O_1$ could be aldehydes and ketones, R+DB $2/O_1$ unsaturated aldehydes, R+DB $4/O_1$ phenols, R+DB $6/O_2$ hydroxy indanones, etc.

Combustor Test Program

Preliminary combustor experiments were carried out with inlet air parameters approximating an idle condition and varying the fuel/air ratio. In order to determine the effects and possible interactions among these parameters on odor intensity and character, all combinations of the four operating parameters were scheduled as test points. The prescribed conditions were as follows:

Inlet air temperature	$^{\circ}C$	40	240
Inlet air pressure	atm.	1	4
Air reference velocity	m/sec.	15	30
Fuel/air ratio		.008	.016

Of the 16 possible test points, we were unable to operate the combustor at six of the conditions, primarily because of low fuel flows and poor fuel atomization.

The test points selected for studying the five fuel variables and the three combustor design variables could be limited to two (or three) cases. These were as follows:

Inlet air temperature	$^{\circ}C$	150	150	240
Inlet air pressure	atm.	2	2	4
Air reference velocity	m/sec.	15	15	15
Fuel/air ratio		.008	.016	.014

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TABLE 4

HIGH-RESOLUTION MASS SPECTROMETRIC

ANALYSIS OF

OXYGENATE FRACTIONS (LCO)

(where ASTM-A1 fuel was burned)

+ R&DB	Number of Oxygen Atoms		Number of Oxygen Atoms	
	O_1	O_2	O_1	O_2
1	1	1	1	0
2	16	6	18	5
3	12	5	19	3
4	11	3	9	2
5	9	4	15	3
6	9	5	11	2
7	5	3	3	1
8	4	2	2	1
Mass	8 µg/ℓ		33 µg/ℓ	
Odor	1.3 TIA		1.95 TIA	
CEFF	99.0%		90.0%	

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Because of the unexpectedly high efficiencies, additional tests were carried out with the inlet air temperatures reduced to 95°C and 10-40°C (ambient air temperature).

Each test point was maintained for 60-90 minutes to allow for sample collection and odor analyses. The observed operating conditions and exhaust analyses were summarized as the average of 2-6 test points recorded in the data acquisition system. With few exceptions, it was possible to complete three test conditions on each test day and generally each condition was replicated on the second test day. The order of presentation was not randomized, but was dictated by the facility scheduling and operational convenience.

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IV. RESULTS

As indicated in the Introduction, the experimental studies are divided into three groups of experiments. The first group of studies was to demonstrate the applicability of the techniques developed in the diesel exhaust program to the analysis of jet combustor exhaust. The second group of experiments was an odor survey on the effect of combustor operating parameters on the odor level and character of the exhaust. The third group of experiments was conducted primarily to determine the effect of combustor modifications and fuel variables on the odor of the exhaust. The results from each group of experiments in the program logically proceeded to the next set of experiments with assurance of a high probability of success.

Preliminary Testing

Early in the program, it was deemed essential to demonstrate the validity of using the methods developed for collection and analysis of diesel exhaust for sampling and odor/chemical analysis of jet combustor exhaust. Therefore, two test points were sampled prior to the installation of the on-line monitoring equipment. These two conditions approximated the idle condition for a jet engine and differed only in the fuel/air ratio. Duplicate preparative-scale collections were made at each test point and taken to Cambridge for extraction and analysis by the ALC. Preparative-scale separation was carried out by liquid chromatography to provide the LCO fraction for high-resolution mass spectrometric (HRMS) analysis. Odor studies were carried out with the TOE. These analytical results are found in Table 5.

TABLE 5

OPERATING CONDITIONS AND EXHAUST ANALYSES

	Operating Conditions				Exhaust Components		
Sample Code	T_i (^oC)	V_r (m/sec)	P_i (atm. abs.)	f/a	TOE ($\mu g/\ell$)	LCA ($\mu g/\ell$)	LCO ($\mu g/\ell$)
NA-1	150	15	2	.008	180	22	25
NA-2					196	29	24
NA-3	150	15	2	.013	81	10	14
NA-4					64	13	15

The TOE abundance was higher with the lower fuel/air ratio averaging 190 $\mu g/\ell$ at 0.008, and 72 $\mu g/\ell$ at 0.013. Similarly, differences were found in the LCA and the LCO fractions with particularly good agreement between the two duplicate samples.

A larger aliquot of the TOE was separated by preparative-scale liquid chromatography to provide an LCO fraction for HRMS analysis. From this analysis, it appeared that the relative distribution of the species containing 1, 2 or 3 oxygen atoms per molecule, and 1 to 8 degrees of hydrogen unsaturation was approximately the same as that found in similar analyses of diesel exhaust extracts. No unique species appeared from the jet combustor samples and, indeed, there were fewer O_3 species throughout the set. This finding gave us confidence that the chemistry of jet combustor exhaust was not unique and, therefore, the detailed analytical information obtained during three years of work with diesel exhaust was directly applicable.

Preliminary odor studies were carried out in the static odor test room in Cambridge at exhaust aliquot concentrations routinely used for the diesel studies, i.e., 20 liters of exhaust in a test room volume of 12,000 liters, a dilution of 600/1, or a concentration of 1.6 ℓ/m^3 of dilution air. The results of these odor studies indicate a higher total intensity of aroma (TIA) for the fraction obtained from the exhaust at 0.008 fuel/air ratio than for the 0.013 fuel/air ratio. Both exhaust samples were noticeably higher in TIA than the reported values for the diesel exhaust at a comparable dilution. Also it was noted that the panel did not generally describe the presence of kerosene (fuel-related) aromatics usually associated with the diesel exhaust. This is consistent with the finding that the LCA fraction was indeed lower than normally found with diesel exhaust.

Odor Survey of the Effects of Combustor Operating Parameters

This group of experiments had the dual function of training the new NASA odor panel and determining those operating parameters which produced recognizable changes in the odor intensity, in order to simplify the test program for the final group of experiments. The test schedule was set up to examine the two extremes for each operating parameter (T_i, V_r, P_i, f/a) in all combinations. This made a total of 16 test points which were scheduled for four test days, i.e., four test points per day. This proved to be an ambitious objective and actually only three test points were attained in each of the test days. However, several of the proposed test points were not feasible to operate and, as a result, 12 tests were carried out which represented 11 different test conditions. These results appear in Table 6, with the operating conditions, odor intensity for the ordered presentation, efficiency,

TABLE 6

OPERATING CONDITIONS AND EXHAUST ANALYSES

ASTM-A1 FUEL/DUPLEX NOZZLE

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ 1ℓ/m³	CEFF	THC µg/ℓ	LCA µg/ℓ	LCO µg/ℓ
*101	30	15	4	.008	41	1.5	71.1	3100	134	64
102	45	16	4	.008	41	2.0	87.8	1100	52	46
103	45	16	4	.016	82	1.75	97.0	370	37	54
104	35	14	1	.013	18	2.0	73.2	2300	138	74
105	35	14	1	.016	23	2.05	79.7	2600	146	104
106	160	17	1	.015	14	2.1	82.4	2000	166	157
107	25	37	1.5	.012	59	1.95	67.5	3300	299	139
108	105	28	1	.017	27	1.9	96.8	475	116	68
109	200	27	1	.009	14	1.95	84.7	985	61	22
110	230	37	3	.016	100	1.3	99.0	35	26	15
111	225	15	4	.016	50	1.4	99.7	10	11	8
112	225	15	4	.009	27	1.25	98.7	22	16	14

* Data from Test 101 not used in correlation.

and exhaust analyses listed. Odor analyses were carried out at five concentrations in duplicate presentations, first ordered and then random. Analytical samples were collected at all test points in duplicate with the exception of those test points in which a preparative sample was collected. The results from Test 101, the first experiment, were excluded from the analysis of the data since the panelists' responses were incon- sistent.

The exhaust hydrocarbon levels varied from over 3000 µg/ℓ for the low efficiency condition to 10 µg/ℓ for the highest efficiency operating condition. The LCA ranged from a high 300 µg/ℓ to a low of 11 µg/ℓ, generally in a similar relationship with the total hydrocarbons. The LCO varied from a high of 160 µg/ℓ to a low of 8 µg/ℓ. The three sets of data were generally parallel in these experiments. The data can be separated into two levels of intensity and two levels of LCO. Those in the range of 2 TIA units are associated with values 40 µg/ℓ of LCO or above and those values below 1.5 TIA units are associated with levels of LCO of 10 µg/ℓ or less. The low odor intensity values are associated with the efficiencies of about 99%.

The set of data can be summarized by plotting the total intensity of aroma (TIA) vs the log of the LCO concentration in µg/ℓ for each test point. These data appear in Figure 11. The least-squares line for these data is computed as $TIA = 0.73 + 0.64 \, logC_{LCO}$. The analysis of the data indicates that a straight line is indeed the best fit, with no quadratic, cubic, or quartic components of significance. Surprisingly, the variability (as indicated by 2σ) is $\pm$ 0.25 TIA units or significantly less than the scale measure used by the panel to record intensity units.

Semi-log correlations are also observed between TIA and concentrations of LCA and THC. The respective curves are $TIA = 0.77 + 0.57 \, logC_{LCA}$ and $TIA = 0.48 + 0.47 \, logC_{THC}$. In both instances, the slopes are lower than the relationship with LCO. The importance of this factor is the observa- tion that, in the dose/response relationship between TIA and exhaust, the values tend to fall between 0.8 and 1.5 with the majority of the slopes above 1.0. If the correlation is to hold that LCO is a measure of TIA, then the slope of this analytical measure should approximate the slope of the exhaust curve.

Quite independently, but also of importance, is the fact that when LCA and LCO are separated by liquid chromatography and presented to odor panelists in Cambridge, the TIA and the character of the LCO fraction most closely approximates the values obtained for the exhaust (or TOE which represents the exhaust). Although LCA has odor at the concentra- tions examined, its intensity does not apparently influence the TIA unless the concentration is as much as 10 times the value of LCO.

Of most importance from this group of experiments is the observation that the panel can only differentiate two sets of inlet-operating con- ditions; those represented by the high-efficiency operation, i.e., T_i of 240°C and P_i of 4 atm., as compared with the inlet air conditions

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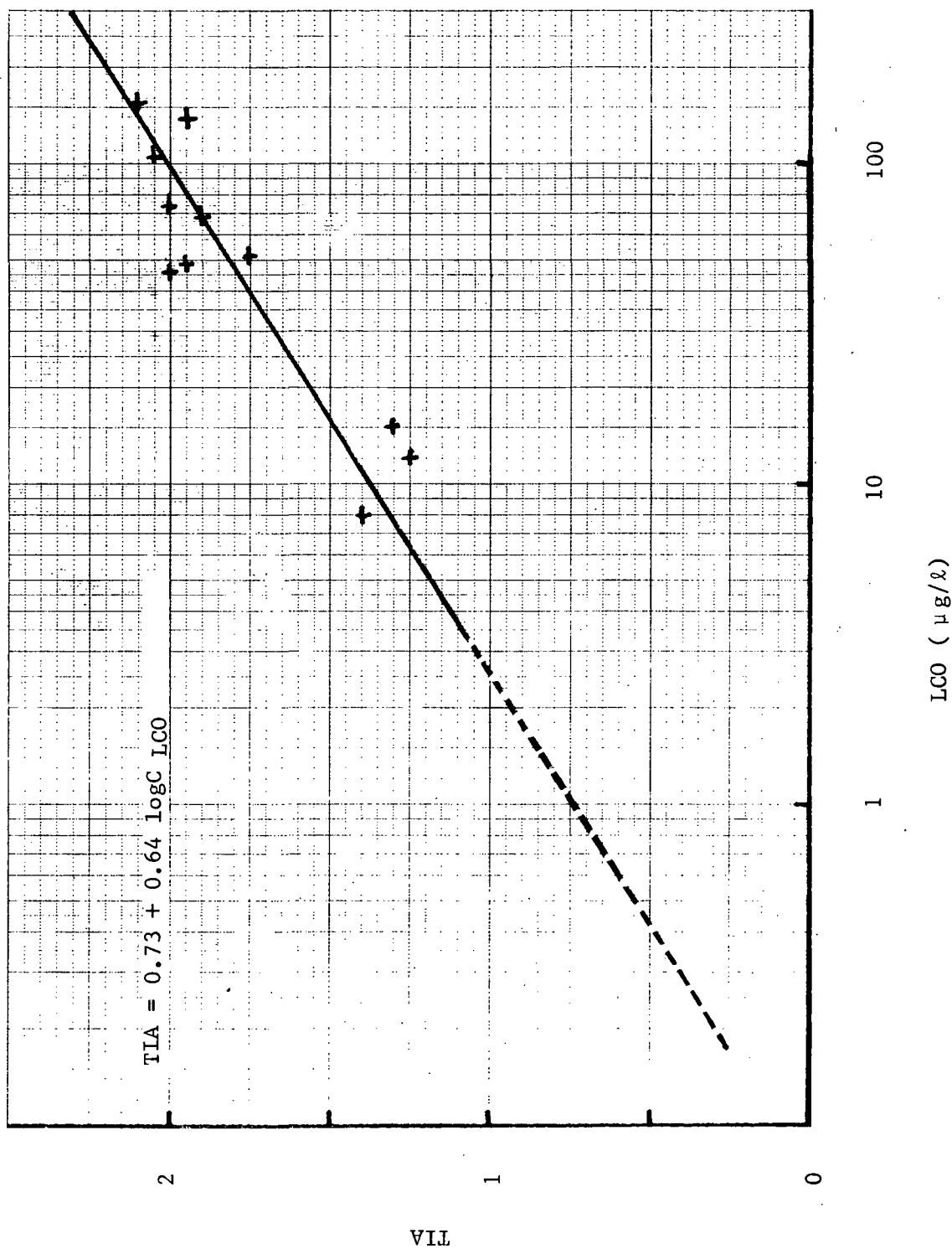
FIGURE 11 TIA vs μg/LCO FOR ODOR SURVEY – ASTM–A1 FUEL/DUPLEX NOZZLE

$$TIA = 0.73 + 0.64 \log_C LCO$$

LCO (μg/ℓ)

TIA

2

1

0

1

10

100

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representing low efficiency idle, i.e., T_i of 150°C and P_i of 2 atm. Therefore, only these two test conditions had to be represented in comparisons of nozzle modifications and fuel variations in further studies.

The second important finding was the fine performance of the NASA panel during this period.

Effects of Combustor Modification
and Operating Variables

The third group of experiments was divided into two sets: those relating to combustor modifications and those relating to fuel variables. The analyses of these sets were treated separately. Two nozzle variations were compared with the performance of the standard Duplex nozzle at three operating conditions.

These conditions were idle and high-efficiency test points, as well as an intermediary condition, which from the original experiment was possibly different in odor intensity from idle. The intermediary condition was constituted by an increase in the fuel/air ratio from 0.008 to 0.016. This proved to be fortuitous since it provided some interesting changes in the odor performance. In addition, with the Duplex nozzle the inlet temperature was studied at two lower temperatures (approximately 30° and 100°C).

Duplex nozzle

The operating conditions, odor and analytical data, for the Duplex nozzle appear in Table 7. As expected, the values for the high efficiency condition produced TIA's below 1.5 and LCO levels below 10 µg/l, while all other conditions produced TIA values in the range of 1.6 to 2.0 and LCO levels from 16 µg/l to a high of 80 µg/l. These values were consistent with July experiments, though there is generally a lower level of LCO produced, which is also reflected in the slightly higher efficiency of operation and the lower TIA values.

The surprising result was observed in Test 208 in which the odor level was essentially as high for the 0.016 fuel/air ratio as the values recorded for the 0.008 condition and the emission level was significantly higher at 80 µg/l of LCO. Operating difficulties encountered during one test day did not permit this condition to be replicated. At this intermediate condition, it was observed that the fuel flow had been split between the primary and secondary nozzle orifices. When the experiment was replicated with all of the fuel flowing through the primary nozzle at the same inlet air condition, the effect was to substantially

TABLE 7

OPERATING CONDITIONS AND EXHAUST ANALYSES

ASTM-A1 FUEL/DUPLEX NOZZLE

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ $1\ell/m^3$	CEFF	THC $\mu g/\ell$	LCA $\mu g/\ell$	LCO $\mu g/\ell$
201	10	14	2	.008	20	1.65	85.7	760	135	16
202	25	14	2	.008	20	1.85	86.4	1300	153	18
203	100	16	2	.008	18	1.75	93.3	410	47	35
204	90	16	2	.008	18	1.85	92.3	470	50	40
205	95	16	2	.016	34	1.61	98.0	150	20	20
206	155	16	2	.008	15	2.05	93.4	360	42	24
207	150	17	2	.008	16	1.8	93.5	410	28	17
208	145	17	2	.016	32	1.8	92.0	1300	127	80
209	240	15	4	.016	50	1.45	99.2	55	5	7
210	240	15	4	.016	50	1.3	99.5	40	3	6

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reduce the odor level to 1.2 TIA units and to reduce the LCO levels to an average of 16 µg/ℓ. These data are tabulated in Table 8. The odor intensities for the idle condition are somewhat lower (1.5) than those previously observed (Tests 206 and 207), although the LCO levels are approximately the same. The value for the high-efficiency level is somewhat higher (1.4) than might be expected for the value of LCO observed, (8 µg/ℓ). These data are apparently inconsistent in as much as lower intensity of aroma was observed for the intermediate test condition with higher LCO values as compared with the high-efficiency test condition.

Air assist nozzle

The first nozzle modification examined was produced by operating the Duplex nozzle with air assist on the alternate orifice. The data for these tests appear in Table 9. The fuel entered through the primary orifice at the two low-temperature conditions, and through the secondary orifice at the high inlet conditions. In all cases, the odor level was below 1.5 and the values for LCO were in the range of 3-6 µg/ℓ of exhaust. The levels for LCA were also in that range and generally lower than in previous data sets. Total hydrocarbon level was also reduced to 50 µg/ℓ, or below, and all of the efficiencies were measured as being 99% or above.

This appears to be a significant improvement in the odorous levels from the jet combustor.

Simplex nozzle

Based on the assumption that spray pattern, or droplet size, could have a significant influence on the odor intensity of the exhaust a Simplex fuel nozzle was installed in place of the duplex nozzle. The data for these tests appear in Table 10. The orifice size was selected to accomodate the highest fuel flow with the available pump pressure, and, therefore, was oversized for the low fuel flow, with the result that high TIA's averaging 2.1, were observed for the idle conditions. These values are at least as high, if not higher, than for any Duplex nozzle operation, however, the LCO values were 30-40 µg/ℓ. The levels at the high efficiency were in the range of 2 to 6 µg/ℓ, and the intensities of aroma below 1.0. Interestingly the intermediate condition was also found to be relatively low in TIA although the levels of 14-16 µg/ℓ of LCO were consistent with those observed for the Duplex nozzle operation with the full flow through the primary orifice.

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TABLE 8

OPERATING CONDITIONS AND EXHAUST ANALYSES

ASTM-A1 FUEL/DUPLEX NOZZLE*

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ $1\ell/m^3$	CEFF	THC $\mu g/\ell$	LCA $\mu g/\ell$	LCO $\mu g/\ell$
501	145	16	2	.008	16	1.5	89.6	680	48	28
502	150	16	2	.008	15	1.55	90.3	580	44	32
503	145	16	2	.016	31	1.2	98.6	90	13	18
504	150	16	2	.015	31	1.25	98.7	68	15	14
505	240	16	4	.014	43	1.4	99.3	32	6	8

* Repeat of 200 series conditions except 503 and 504 fuel flow is all through primary as compared with 208.

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TABLE 9

OPERATING CONDITIONS AND EXHAUST ANALYSES

ASTM-A1 FUEL/AIR ASSIST NOZZLE

Test Code	Operating Conditions T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	Odor Intensity TIA @ $1\ell/m^3$	Efficiency CEFF	Exhaust Components THC $\mu g/\ell$	LCA $\mu g/\ell$	LCO $\mu g/\ell$
401	150	17	2	.008	17	1.25	99.0	30	2	3
402	160	16	2	.009	17	1.3	99.3	30	1	2
403	150	17	2	.016	33	1.25	99.0	55	4	6
404	160	16	2	.017	33	1.4	99.3	30	4	6
405	240	16	4	.013	42	1.35	99.1	45	3	4
406	245	16	4	.013	42	1.05	99.2	30	2	6

TABLE 10

OPERATING CONDITIONS AND EXHAUST ANALYSES

ASTM-A1 FUEL/SIMPLEX NOZZLE

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ $1\ell/m^3$	CEFF	THC $\mu g/\ell$	LCA $\mu g/\ell$	LCO $\mu g/\ell$
701	145	16	2	.008	16	2.25	83.1	1160	175	37
702	145	17	2	.008	16	1.95	86.7	860	138	32
703	145	16	2	.016	32	1.15	99.1	35	22	14
704	140	16	2	.016	32	1.15	98.9	55	26	16
705	235	15	4	.014	44	.8	99.9	1	2	2
706	235	15	4	.014	44	.7	99.8	2	4	6

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Fuel Variables

Four fuels, in addition to ASTM-A1 fuel, were examined in the course of the program: natural gas with no aromatics and a gas at ambient temperatures, isooctane, a hydrocarbon, with essentially no aromatics, JP-4 with slightly higher aromatics, and JP-5 type, a specially blended fuel, an aromatics concentration of 30%. The 50% boiling points (100% for isooctane) are listed below:

ASTM-A1	–	$205^{\circ}C$
JP-5 type	–	$230^{\circ}C$
JP-4	–	$150^{\circ}C$
Iso-octane	–	$116^{\circ}C$ (B.P.)

Each of the four fuels was examined at the three standard test points, and each test replicated on the second day. With the exception of the natural gas, an additional test point was examined in which the inlet air temperature was reduced to between 10 and $50^{\circ}C$.

Natural gas

As might be expected, the natural gas gave the lowest total intensity of aroma. As shown in Table 11, all TIA values were below 0.7 and in the highest efficiency condition in one of the tests, the value was calculated to be 0.1, which is slightly below the value found for background odor level of the test room, although the character was recognizable as burnt. Indeed, in order to measure the intensity of aroma from this series of samples, the concentration range was increased to a maximum, i.e., a dilution of only 35/1 at the highest level. This would suggest recording TIA for 10ℓ of exhaust per m^3 which indeed eliminates any background. Values for LCO were measured in all samples analyzed. The initial test results were apparently inflated by contamination from previous test runs. In other cases, values from 1.3 to 0.5 $\mu g/\ell$ of LCO were measured. These were generally commensurate with the intensities of aroma observed in this series of samples, although they are near the threshold for measurement with the analytical system and standard sample size used in these experiments.

Isooctane

Isooctane also produced uniformly low odors at all of the three standard test conditions with the values for TIA near 1.0 and LCO values of 3-4 $\mu g/\ell$. The exception was the lowest temperature condition, air inlet temperature of $10^{\circ}C$, where the TIA observed as 1.6, and the LCO value was increased to 10 $\mu g/\ell$. These data appear in Table 12.

TABLE 11

OPERATING CONDITIONS AND EXHAUST ANALYSES

NATURAL GAS FUEL/SPECIAL NOZZLES

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ $1\ell/m^3$	CEFF	THC $\mu g/\ell$	LCA* $\mu g/\ell$	LCO* $\mu g/\ell$
901	145	17	2	.008	16	.45	95.2	260	ND	0.5
902	145	15	2	.008	15	.45	96.8	190	NA	NA
903	145	17	2	.015	31	.7	97.2	260	ND	.8
904	145	16	2	.016	31	.3	98.0	170	.1	1.2
905	235	15	4	.013	40	.35	99.8	10	**	**
906	235	15	4	.012	39	.1	99.8	7	ND	1.3

*   System blank 0.8 and 0.7 µg/ℓ respectively subtracted.
** 5.0 and 9.0 µg/ℓ respectively represents probable system contamination.
ND Not Detected.
NA Not Analyzed.

TABLE 12

OPERATING CONDITIONS AND EXHAUST ANALYSES

ISOOCTANE FUEL/DUPLEX NOZZLE

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ $1\ell/m^3$	CEFF	THC µg/ℓ	LCA µg/ℓ	LCO µg/ℓ
601	10	14	2	.008	19	1.6	89.2	1180	62	10.
602	150	16	2	.008	14	.9	97.2	150	2	4
603	145	16	2	.008	15	1.0	97.1	160	1	4
604	145	16	2	.015	26	1.1	98.3	110	2	3
605	145	16	2	.015	27	.8	98.1	120	2	3
606	235	16	4	.012	36	1.0	99.2	48	2	4
607	235	15	4	.013	36	1.0	99.4	40	2	4

JP-4

With JP-4, TIA values for all of the standard conditions fell in the range of 1.4-1.6 TIA units. A possible anomaly in the data appeared at the intermediate condition where the concentration of LCO increased four fold to 35-38 μg/ℓ, with no measured change in the TIA. Curiously, at the low inlet air condition, 40°C, the TIA in both instances was 1.8, with an LCO level of approximately 13 μg/ℓ. These data appear in Table 13.

JP-5 type

The data for JP-5 type fuel appears in Table 14. The odor levels from JP-5 were somewhat higher in all instances than the values observed with other fuels, including ASTM-A1 at the standard operating condition, while the LCO measurements were in the range of 5 to 10 μg/ℓ. The one exception was the high efficiency test condition where the run was aborted midway through. This may have influenced the hydrocarbon level during part of the collection. TIA values for the inefficient condition were 2.0, but were associated with only 10 μg/ℓ of LCO.

An interesting observation here is that the least-squares line for these values approximates quite well the line determined for diesel exhaust. (TIA $-$ 1.1 $+$ 0.9 $\log C_{LCO}$ vs TIA $=$ 1.0 $+$ 1.0 $\log C_{LCO}$ for diesel). Indeed this fuel was designed to represent a commercial-type diesel fuel. With the low inlet air temperature, the LCO value increased to 24 μg/ℓ, while the TIA measure was 1.8 which is consistent with the values measured for the other fuels at this reduced efficiency condition.

Odor Character and Composition Variation

The primary odor characteristic of all of the exhaust samples is a smoky or burnt-smoky odor. With the low aromatic fuels the odor type is more sweet and burnt rather than smoky and tarry that is perceived with the higher aromatic fuels. In general, there is little fuel-related character in combustor exhaust. When it appeared it was usually associated with low efficiency conditions and most frequently with JP-5 and JP-4. A solventy odor, characteristic of the isooctane itself, was detected in the exhaust produced with that fuel. Odor profile composite descriptions of the exhaust from the five fuels appear in Table 15. Pungency or with JP-4, nose irritation was apparent in all samples. An oily note was perceived in all samples but was described as oxidized oil only in those fuels that contained significant levels of aromatics.

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TABLE 13

OPERATING CONDITIONS AND EXHAUST ANALYSES

JP-4 FUEL/DUPLEX NOZZLE

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ $1\ell/m^3$	CEFF	THC $\mu g/\ell$	LCA $\mu g/\ell$	LCO $\mu g/\ell$
301	10	14	2	.008	19	1.85	89.0	1160	95	14
302	40	14	2	.008	19	1.80	90.9	800	83	13
303	145	17	2	.008	16	1.3	96.1	270	16	12
304	150	17	2	.008	16	1.5	96.3	260	14	8
305	150	17	2	.016	32	1.6	95.2	690	65	35
306	145	17	2	.015	33	1.4	94.4	780	72	38
307	235	15	4	.013	42	1.5	98.9	90	7	8
308	240	15	4	.013	42	1.4	98.6	90	6	8

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TABLE 14

OPERATING CONDITIONS AND EXHAUST ANALYSES

JP-5 TYPE* FUEL/DUPLEX NOZZLE

Test Code	Operating Conditions					Odor Intensity	Efficiency	Exhaust Components		
	T_i °C	V_r m/sec	P_i atm	f/a	Fuel Flow g/sec	TIA @ 1ℓ/m³	CEFF	THC µg/ℓ	LCA µg/ℓ	LCO µg/ℓ
1001	30	14	2	.008	21	1.8	85.9	690	206	24
1002	145	16	2	.007	15	2.0	93.6	500	54	10
1003	145	16	2	.008	15	1.9	93.5	500	54	10
1004	150	16	2	.015	30	1.75	97.8	114	25	6
1005	235	15	4	.014	43	1.6	98.2	105	15	5
1006**	240	15	4	.014	42	1.6	97.5	185	45	15

*Specially blended fuel - 30% aromatics.

** Test aborted.

TABLE 15

ODOR PROFILE COMPOSITES

EXHAUST CHARACTER ASSOCIATED WITH FUEL TYPE

	Natural Gas	Isooctane	ASTM-A1	JP-4	JP-5
	Burnt Sweet	Burnt Smoky	Smoky Burnt	Burnt Smoky	Smoky (Burnt Tarry)
	Sooty	Oily	Oily Oxidized	Kerosene	Kerosene
	Oily (soapy)	Solvent	Sweet Solvent	Oily Oxidized	(low efficiency)
	Pungent	Pungent	Pungent	(leather)	Sour Oily
		Sour (yeasty)		Nose Irritation	Slight Pungent
TIA_{found}	.35/µg LCO	.44/µg LCO	.73/µg LCO	1.36/µg LCO	1.68/µg LCO
$TIA_{calc.}$	.35/µg LCO	.47/µg LCO	.44/µg LCO	.63/µg LCO	.88/µg LCO

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V. DISCUSSION OF RESULTS

As a result of the experimental studies, it is clear that inlet operating parameters, fuel types and combustor modifications can all influence the intensity of exhaust odor calculated for a standard concentration of exhaust in dilution air. Associated with the changes in odor intensity are changes in exhaust composition and combustor operating efficiency. The experimental results have been examined independently and in combination to determine which of these measures, expressed logarithmetically, might be most useful for estimating the sensory response. An important consideration in selecting analytical measures for correlation with odor intensity is the relationship between the measured species and the sensory response. For this reason, our work has emphasized the correlation between odor intensity and the concentration of the partial combustion products as measured by LCO.

Because of the practical value of a correlation between odor and operating efficiency, this data for tests with ASTM Al fuel is included. An expression which represents inefficiency of combustor operation (100-CEFF) has been used to graphically present this relationship. This may also be represented by the milligrams of LCO per gram of fuel burned.

Finally, in evaluating the environmental impact of combustor exhaust, the total quantity of odorous material emitted during a prescribed time determines the "odor load" on the diluting atmosphere. This is the product of the odor level of the exhaust (LCO) and the volume of exhaust emitted per unit time.

Odor Analytical Correlations

Including the data from all tests carried out under this program, the expression for odor strength is found to be:

$$TIA = 0.68 + 0.72 \ logC_{LCO}$$
$$2\sigma = 0.46$$
$$r^2 + 0.67$$

The correlation of TIA with LCA is at least as good for all experiments, except, of course, for those in which natural gas was the fuel since no measurable LCA was found.

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The correlation can be improved by the inclusion of the on-line exhaust analytical data (represented by CEFF & THC). Combustion inefficiency (100 -CEFF) gives a slightly more significant result than total hydrocarbon data. Including this measure in the regression analysis leads to the expression;

$$TIA = 0.75 \times 0.55 \, \log C_{LCO} + 0.23 \, \log \, (100 - CEFF)$$

With this addition the correlation as measured by r^2 is increased only to 0.72.

Comparing the correlations for the ordered presentations with the data from the random presentations had the effect of reducing the TIA intercept by 0.1 TIA units. Since the inclusion of the random data did not affect the conclusions and increased the variability, they have not been used in the final correlations. Comparison of the analytical results from the prep scale samples and the analytical scale samples shows the prep scale to be about 15% higher on the average. However, the correlations of TIA with each set of samples independently are not significantly different. Therefore, in the analyses all analytical values for each test point have been averaged.

Combustor Variables

During the last phase of the program, odor data associated with experiments using ASTM-A1 fuel and with variations in the nozzle operations were compiled. They are summarized in Table 16. From Figure 12 it can be seen that this spread in values extends the range of concentrations studied by one order of magnitude as a result of the exhaust from the air assist and Simplex nozzle modifications. The calculated line;

$$TIA = 0.79 + 0.60 \, \log C_{LCO}$$
$$2\sigma = 0.46$$
$$r^2 = 0.58$$

is almost exactly the same as that determined for the odor survey data.

It can be seen from the data that the air assist nozzle reduced both the odor level and the concentration of oxygenates in all three test conditions, but most significantly in the low-efficiency condition where there is an order of magnitude reduction which is associated with a 0.5 TIA unit decrease in the intensity of odor. The Simplex nozzle also produced a significant reduction in the odor at the high fuel flow condition, but was high in odor with low fuel flow. An interesting observation which emerged from the first of experiments was the fact that in the intermediate test condition, $150^{\circ}C - 0.016$ fuel/air ratio, when the fuel flow was split between the primary and secondary nozzles, the efficiency was lower and the odor intensity as well as concentration of oxygenates were substantially higher.

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TABLE 16

TIA INTERCEPT

AND

OBSERVED LCO VALUES

FOR

COMBUSTOR MODIFICATIONS AT VARIOUS INLET AIR TEMPERATURES

Test Condition		Nozzle Operation							
		Duplex		Air Assist		Simplex		Primary/Secondary	
T_i °C	f/a	TIA $1\ell/m^3$	LCO $\mu g/\ell$	TIA $1\ell/m^3$	LCO $\mu g/\ell$	TIA $1\ell/m^3$	LCO $\mu g/\ell$	TIA $1\ell/m^3$	LCO $\mu g/\ell$
150	008	1.71	25.2	1.3	2.5	2.1	34.5		
240	016	1.4	6.5	1.2	5.0	-	-		
240	014	1.4	8.	1.3	6.0	.75	4.0		
150	016	1.2	16.0	-	-	1.15	15.0	1.8	80.0
95	008	1.8	37.5						
95	016	1.6	20.0						
40	008	1.75	17.0						

+ Standard
x Air Assist
o Simplex
□ Primary/
  Secondary

$TIA = 0.79 + 0.60 \log C_{LCO}$

10

1

LCO ($\mu g/\ell$)

TIA

2

1

0

FIGURE 12   TIA vs μg/LCO FOR ALL NOZZLE VARIATIONS

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Fuel Variables

The second set of data from these experiments was the comparison of the odor levels from four different test conditions for five different fuels reported in Table 17. It was apparent that as the volatility of the fuel increased from ASTM-A1 to natural gas, there was a concurrent reduction in the intensity of odor at all of the exhaust conditions, with an associated reduction in the concentration of the oxygenate species. Natural gas, surprisingly, does produce odor but at very low intensities when examined at 1000/1 dilutions. We were able to measure concentrations of the oxygenates in these samples. The reproducibility is not as good at these levels because of low LCO values and the interference of background. The correlation found between TIA and LCO when the data for all five fuels at similar operating conditions were analyzed is:

$$TIA = 0.59 + 0.89 \ logC_{LCO}$$
$$2\sigma = 0.55$$
$$r^2 = 0.74$$

This line has a higher slope and lower intercept than for all tests with ASTM-A1 fuel. The curve of TIA vs $logC_{LCO}$ appears in Figure 13. When all of the data from all of the experiments is pooled, however, there does not appear to be a significant difference in the relationship between TIA and LCO for the several fuels.

If the TIA intercepts and the slopes for the individual fuels are com-[*] pared with the values for all fuels combined, one can use the "t" test[*] to measure significance of the differences. No significant differences are found between the various slopes. There is a 95% probability that the intercept for JP-5 (1.68), Isooctane (0.44), and natural gas (0.35) are significantly different from the ASTM-A1 value (0.73).

Efficiency and Emission Levels

In examining the other parameters which may relate to odor intensity, and which are normally measured in combustor tests, there appears to be a reasonable correlation between the TIA and the log of the inefficiency of combustor operation, represented as 100 -CEFF, and plotted TIA vs its log (Figure 14). Over all of the Task III ASTM-A1 fuel tests, it can be seen that the values fall in three separate groups with highest efficiency observed with the air-assist nozzle operation, and the lowest efficiency observed with the lower inlet air temperature and low fuel/air ratio. The least-squares line for these values, the values representing all of the ASTM-A1 experiments, is TIA = 1.3 + 0.77 log (100 -CEFF). Thus at 99% efficiency, the intensity of odor is approximately 1.3 TIA units at a 1000/1 dilution. When engine operating efficiency is increased to 99.9% the expected intensity of aroma should be approximately 0.5, and indeed it was found to be 0.75 TIA units.

* Students "t" test for statistical significance.

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TABLE - 17

TIA INTERCEPT

AND

OBSERVED LCO VALUES

FOR

VARIOUS FUELS AT FOUR INLET AIR CONDITIONS

Test Condition	T_i 150°C, f/a .008		T_i 240°C, f/a .014		T_i 150°C, f/a .016		T_i 40°C, f/a .008	
Fuel	TIA 1ℓ/m³	LCO μg/ℓ	TIA 1ℓ/m³	LCO μg/ℓ	TIA 1ℓ/m³	LCO μg/ℓ	TIA 1ℓ/m³	LCO μg/ℓ
ASTM-A1	1.7	25.2	1.4	8.0	1.2	16.0	1.75	17.0
JP-4	1.4	10.0	1.45	8.0	1.5	36.5	1.8	13.5
Isooctane	1.0	4.0	1.0	4.0	.95	3.0	1.6	10.0
Natural Gas	.45	.5	.2	1.3	.5	1.0	-	-
JP-5 type	1.95	10.0	1.6	10.0-	1.75	6.0	1.8	24.0

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TIA = 0.59 + 0.89 logC_{LCO}

+ ASTM-Al
x JP-4
o Isooctane
□ Natural gas
△ JP-5

LCO (μg/ℓ)

FIGURE 13 TIA vs μg/LCO FOR ALL FUELS

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$$TIA = 1.3 + 0.77 \log(100 - CEFF)$$
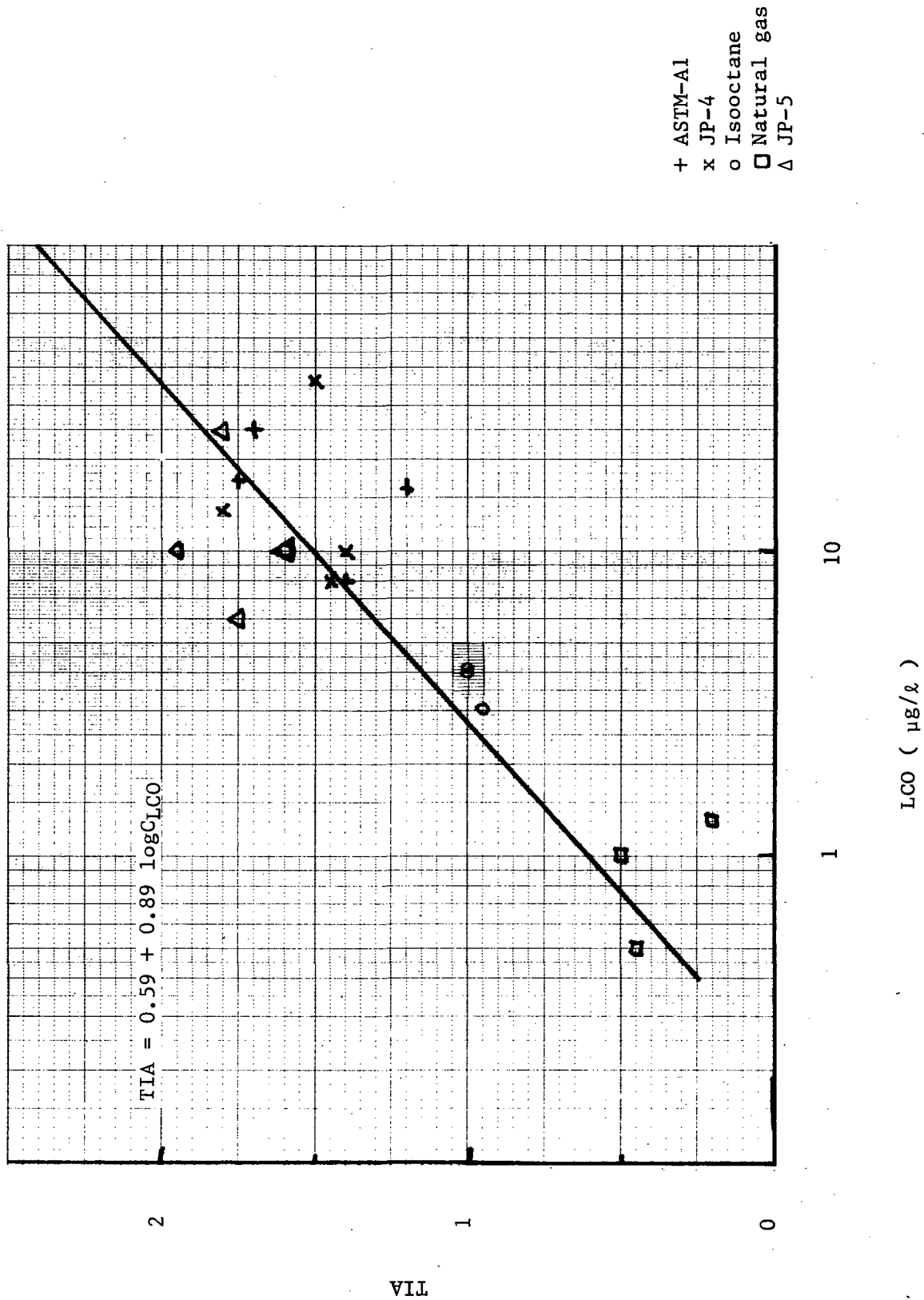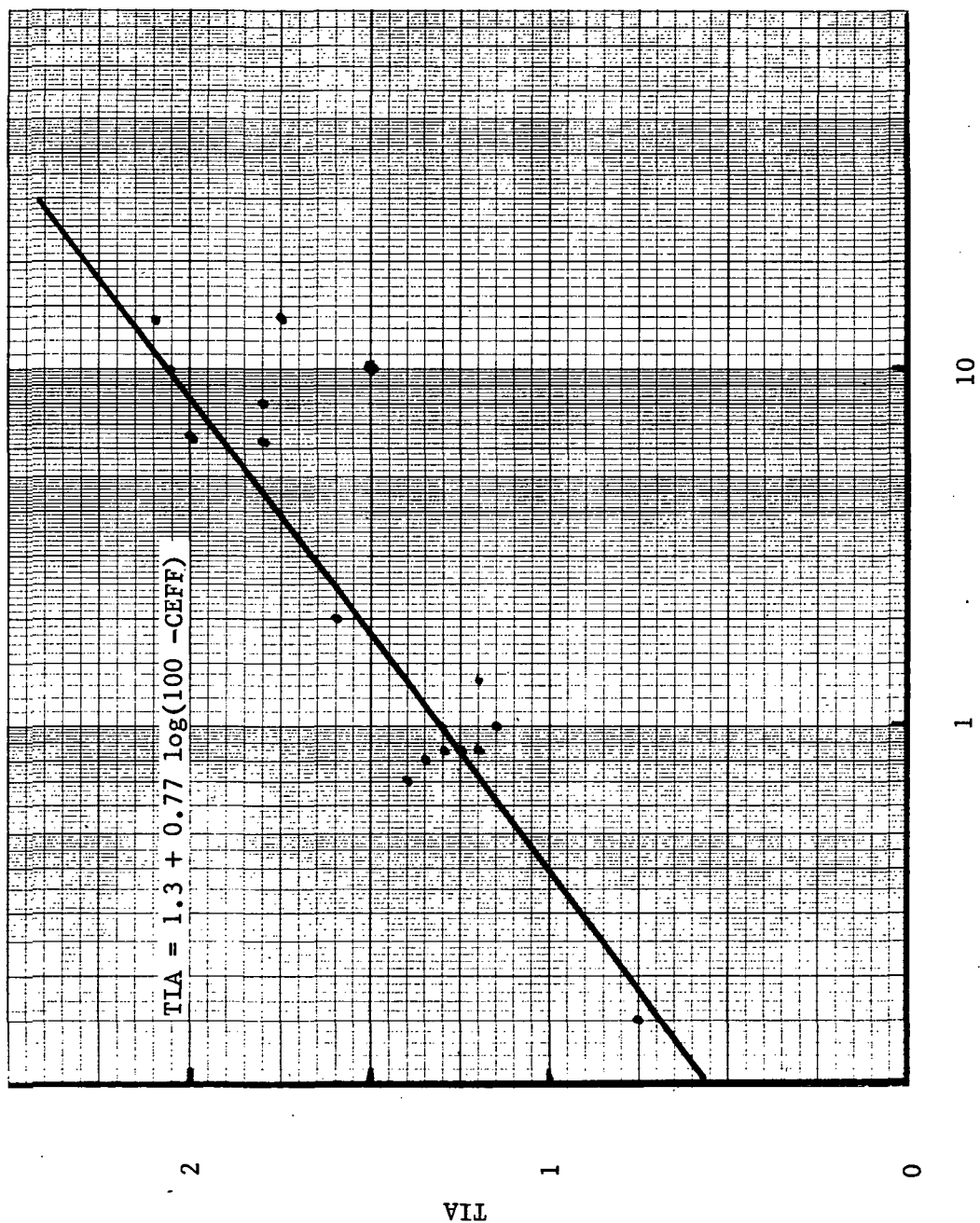
FIGURE 14 TIA vs INEFFICIENCY FOR TESTS WITH ASTM-A1 FUEL

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A similar correlation is found when the data for all fuels are combined. There is, however, considerably more scatter in the data. This would be consistent with the observations that with different fuels different levels of LCO are produced and the hypothesis that the LCO's for different fuels have different values of TIA/μg.

It is apparent that there is also a relationship with efficiency and mg of LCO produced per gram of fuel (ASTM-Al) burned. At high efficiency (about 99% CEFF) the calculated values are 1.0 mg/LCO/g of fuel (0.1%) or less. The maximum value 10.5 mg/g fuel was found with a very low efficiency of 67.5%, while the next highest value of 9.4 mg/g at 81.4% CEFF. The values calculated for a series of ASTM-Al fuel tests are given in Table 18.

Although the milligram percent of odorant produced for each gram of fuel burned has an influence on the impact of jet combustor exhaust on the environment, the calculated total quantity of odorant emitted per unit time is probably more important. This is the product of the air flow (m^3/sec) and the odorant load (mg LCO/m^3).

The values cover two orders of magnitude from 600 mg/sec at low efficiency and high fuel flow to 4 mg/sec at idle with the air-assist nozzle.

Even at the high efficiency where both air and fuel flows are high, the odorant load on the environment was found to be 87 mg/sec as compared with the average for several idle condition results of 50 mg/sec.

Odor Character and Composition Variation

Samples from natural gas and from iso-octane are recognizably different in character from the other three fuels. It would also appear that the JP-5 exhaust could also be discriminated because of its sourness and burnt tarry character.

In correlating the odor intensities measured for different fuels with the amount of LCO measured, there are significant (95% confidence) differences between the TIA per μg of LCO observed for ASTM-Al and those observed for natural gas or JP-5. In no cases are the slopes of the dose/response curves sufficiently different. The values are recorded in Table 15 as TIA found, that is the TIA per μg LCO from the observed dose/response curve and TIA calculated which assumes the same slope (0.89) for all fuels.

There do not appear to be significant differences in odor type between low or high efficiency operating conditions for the same fuel. Several test points suggest that irritation factors are more apparent at the low inlet air temperature (150°C and may be higher in strength at the higher fuel/air ratio.

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TABLE 18

EMISSION LEVELS

OF

ODORANT SPECIES

ASTM-A1

Test Code	CEFF %η	mg LCO/ g fuel	Air Flow m^3/sec	LCO mg/sec
107	67.5	10.50	4.29	596
105	79.7	6.86	1.16	140
106	82.4	9.38	.95	149
701/702	84.9	4.03	1.82	64
201/202	86.8	1.94	2.23	38
206/207	93.4	2.23	1.82	36
208	93.7	4.88	1.82	155
503/504	98.6	.94	1.82	29
110	99.0	.85	5.78	87
401/402	99.2	.26	1.78	4
209/210	99.4	.43	2.85	21
705/706	99.8	.26	2.81	11

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The HRMS matrix analysis has been carried out for a total of 16 test points, the majority using ASTM-A1 fuel. Although differences appear between samples, they do not appear to occur consistently. Of more importance, however, is that data is not available for the two fuels which are most different in odor type and intensity from ASTM-A1 fuel. There was not sufficient material collected from the natural gas experiment for analysis and instrumental difficulties have delayed the results for JP-5 fuel.

In addition, the complexity of the matrix, 16 independent variables, and the paucity of observations makes it impossible to draw sound conclusions. A principle components analysis[8] was carried out for eleven matrices, which do not indicate any significant relationships which could be used to simplify the data set. Multiple regression analyses were carried out with TIA's as the dependent variable and although some correlations were observed, the coefficients were no better than with total LCO.

It is clear, however, from these data that the chemistry of combustor exhaust is similar in both types of species present and general chemical complexity to diesel exhaust. Detailed identification studies would probably not produce more useful data than has been obtained.

VI. CONCLUSIONS

The experimental program showed that the odor intensity of jet combustor exhaust is apparently decreased by those inlet operating parameters and combustor modifications which act to increase the burning efficiency as computed from the exhaust gas composition. The partially oxygenated components, as measured by an analytical liquid column chromatographic system, correlate well with the intensity of odor as determined by a defined method of the sensory response and potential environmental effects, regardless of fuel type or combustor operating conditions.

At comparable inlet operating conditions, the type of fuel burnt can have a substantial effect on the perceived odor intensity. This effect is correlated with changes in the concentration of the oxygenates (LCO) determined in the exhaust. Similarly, those combustor modifications which significantly reduce the intensity of odor are those which reduce droplet size and increase the dispersion.

Changes in fuel type are associated primarily with differences in odor character. The ALC system measures only the amount of LCA and LCO but gives no information on the composition of the LCA or LCO fraction. Other techniques such as high resolution mass spectrometry would be required to furnish this information.

The sensory analyses by the newly-trained NASA panel have provided consistent measures of odor. In general, their response is somewhat lower than we have observed with the more experienced ADL panel, which may reflect differences in sample presentation or less experience in intensity discrimination. It is possible, however, to obtain relevant odor information from sensory analyses of the total organic extract from an experienced odor panel.

Recommendations

Since the present study has been limited to the exhaust of a single combustor at one sampling point, it is recommended that the experimental study be extended to determine the effects of turbine operation, design variations, and sampling points on the odor intensity and oxygenate's concentration. Sampling could be carried out at NASA-Lewis or at commercial test stands and odor/chemical analyses carried out at ADL's Cambridge laboratories.

Because of the profound effect of factors which affect the rate of fuel vaporization on the odor intensity and concentration of oxygenates, we recommend an exploratory research investigation to examine in the laboratory the effects of droplet size and particle distribution on odor intensity and LCO concentration in order to understand the potential contribution of combustor design and fuel selection to the reduction of odorous air pollution.

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The primary impact of combustor exhaust is in and around airports.
We recommend that a study program be undertaken to attempt to measure
odor intensities at various airport locations, and collect ambient
samples for laboratory odor and/or chemical analyses in order to esti-
mate whether the predicted odor dilution values are realistic and to
determine the actual dilutions which can be expected under various atmos-
pheric conditions.

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